Investigations Concerning the Thermal Alteration of Silica Minerals: An Archaeological Approach

By

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TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

Abstract of Dissertation Presented to the Graduate Council of the University of Florida in Partial Fulfillment of the Requirements for the Degree of Doctor of Philosophy

INVESTIGATIONS CONCERNING THE THERMAL ALTERATION OF SILICA MINERALS: AN ARCHAEOLOGICAL APPROACH

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A recent publication (Crabtree and Butler 1964) suggests that prehistoric man may have found it advantageous to thermally alter lithic raw materials prior to manufacture or final completion of chipped stone implements.

The specific objectives of this research and the conclusions reached are as follows:

1. To establish whether a functionally desirable change in chert occurs when it is subjected to heat. Experiments were conducted to determine the temperature and length of time necessary to effect the detectable physical alterations. Heated and unheated Florida cherts were studied by petrographic analysis, x-ray diffraction, differential thermal analysis, scanning electron microscope, atomic spectrophotometer analysis, standard rock mechanics test, and the gas absorption surface area and porosity technique.

2. To determine whether primitive peoples might have been aware of the advantages conferred by thermally altering lithic materials. A review of the literature revealed no completely adequate account describing this technique. Enough records exist, however, to warrant the

conclusion that fire was used sometimes in conjunction with lithic technology.

An examination of archaeological chipping debris revealed that a portion of this debitage had been intentionally flaked after heating. The flaked surfaces are lustrous and differ in this respect from outcrop samples of the same materials. The flakes also have bulbs of percussion indicating that impact has taken place. Bulbs of percussion are not present when rocks explode from thermal stresses though the conchoidal fracture typical of flint materials is evident.

3. To recommend a technique which might be employed by archaeologists to determine whether the chipped stone remains recovered from sites had been thermally altered. No practical standardized test was discovered. However, from an examination of a representative sample of flaking debris, an investigator should find a number of specimens exhibiting a relict dull area surrounded by areas of high luster. This situation strongly suggests that the dull area has not been flaked subsequent to heating, whereas the vitreous area has been flaked.

Temperatures of 350°C-400°C are sufficient to alter Florida cherts but x-ray diffraction patterns demonstrated that no change in the crystal lattice occurs. This was borne out by petrographic analysis which showed no change in the shape, size, or orientation of the microcrystals. The alteration is due, at these low temperatures, to the presence of impurities in the rock serving as fluxes to

firmly cement the microcrystals of quartz. This fusion led to approximately a 45% reduction in strength necessary to fracture Florida cherts under point tensile load, and approximately a 60% reduction in the intergranular surface area. When the rock is fractured subsequent to thermal alteration, the fractured surface is extremely vitreous. The fracture splits the grains, rather than breaking around them as in unheated counterparts, revealing the true luster of quartz. Since crystal boundaries are no longer interfering with the removal of flakes, heated material fractures more like glass than like a rock aggregate. This fact is dramatically illustrated by the change in surface topography when viewed by the scanning electron microscope. A color change may occur when cherts are heated if iron is present in sufficient quantities. Color change occurs at a lower temperature than that effecting thermal alteration but may be used as a valid criterion if accompanied by vitreousness.

The information gained from this study leads to the conclusion that if flint materials are cautiously heated for sustained periods, an alteration occurs which confers an advantage in manufacturing chipped stone implements. Furthermore, prehistoric peoples were probably well aware of this advantage.

INTRODUCTION

A recent publication (Crabtree and Butler 1964) suggested that prehistoric man may have found it advantageous to thermally alter his lithic raw materials prior to manufacture or final completion of chipped stone implements. Subsequent examination of chipped stone tools as well as of waste flakes recovered from archaeological sites led to the speculation that this technique had been employed aboriginally in the state of Florida. Florida materials often exhibit the pinkish cast and vitreous luster felt to be indicative of thermal alteration and differ markedly from the siliceous materials found in outcrops.

Since the summer of 1968, the author has studied the collections of stone tools in the Florida State Museum. Specimens from every Florida county are represented but samples are scarce from counties where there are no natural outcrops of chert, e.g., south Florida. Waste flakes were not available since they had been discarded. For several years, however, the Department of Anthropology at the University of Florida has been storing all stone materials recovered, including chipping debris. This debitage was also examined. This extensive search has led to the deduction that the original speculation was valid, i.e., many of the chipped stone remains appear to have been thermally

altered. The number might be greatly increased if it were known whether patinated specimens or specimens recovered from under the water had been subjected to heat. Patination as well as minerals present in Florida lakes, rivers, and springs cause changes which conceal the original texture of chert, making it impossible to determine (at present) if thermal alteration has taken place.

The objectives of the research then undertaken were:

1. To establish whether a functionally desirable change in the chert occurs when it is thermally altered. The experiments conducted are described in the section on Methodology which embodies the major portion of this dissertation.

2. To demonstrate whether prehistoric peoples might have been aware of the advantages conferred by thermally altering their lithic materials. To aid in this endeavor, an intensive and extensive search of existing publications was undertaken. The results of this search are described in the Literature Review.

The use of analogy in interpreting archaeological data "In its most general sense . . . is assaying any belief about nonobserved behavior by referral to observed behavior which is thought to be relevant" (Ascher 1961: 317). Unfortunately, historic accounts were not found which accurately described the process of thermal alteration. However, enough descriptions of the use of fire during some stage of stone tool manufacture were uncovered to warrant the conclusion

that the "observed behavior" would have been "relevant" to the problem being investigated if the observers had paid more attention to detail and had provided a more thorough description.

In addition, in the section on Methodology, comparisons are made between materials that have been intentionally subjected to heat under controlled conditions and subsequently flaked with samples exhibiting potlid fractures where heat has been applied suddenly, e.g., through forest fires. Other types of fracture resulting from expansion and contraction are noted. This analysis served as a reminder that flint materials may break with a conchoidal fracture but that no bulb of percussion will be present unless impact has taken place. The archaeological debitage suspected of being intentionally heated does possess bulbs of percussion. "Solutions to any problem are at best approximations arrived at by the elimination of those least likely" (Ascher 1961: 323). In this case, archaeological remains have provided clues to prehistoric practices through systematic elimination of alternative solutions.

The hypothesis that heat was used by aboriginal peoples to alter lithic raw materials prior to final manufacture of chipped stone tools is strengthened by (1) historic accounts, (2) experimentation with and study of intentional vs unintentional fractured surfaces, (3) comparisons between outcrop and site materials, (4) heating experiments with outcrop materials resulting in specimens whose appearance

resembles artifactual remains, and (5) tests demonstrating that thermally altered siliceous materials are easier to flake.

3. To suggest a technique which might be employed by archaeologists to determine if the chipped stone remains recovered from archaeological sites had been thermally altered. This presupposes that the alteration is a permanent one readily ascertained by subjecting the material to some sort of standard test. This problem is discussed in the section on Archaeological Application.

LITERATURE REVIEW

Following Crabtree and Butler's (1964) article, archaeologists began to look for indications of alteration by heat of materials used in making chipped stone implements. It now appears that this phenomenon is very widespread. Despite the fact that most early explorers, colonists, traders, missionaries, and adventurers were very poor ethnologists, it seemed surprising that this technique had not been observed and described. American Indians, as well as other primitive peoples around the world, readily perceived the advantages of iron tools which were introduced to them through European contact. They willingly put aside nearly two million years of stoneworking technology, often within a single generation. Unfortunately, very little attention was devoted to the material or practical aspects of Indian life until the middle of the nineteenth century. It wasn't until many aboriginal practices had been discontinued that a rapid attempt to record these practices was undertaken. In some cases, this attempt came too late. For obvious reasons, therefore, it was necessary to turn to nineteenth century sources almost entirely. Thus, literature, which in most fields of endeavor has been laid to rest, was reviewed in an attempt to shed some light on a recent observation of man's past behavioral patterns.

Many excellent accounts exist of aboriginal stone-working techniques. In addition, numerous experimental studies have been conducted and reported upon which describe the step by step manufacture of tools by direct percussion, indirect percussion, pressure, and various combinations of these methods. These techniques constituted the major processes employed in shaping and finishing stone tools. The author does not wish to give the impression that it was always necessary to thermally alter lithic materials. Some materials probably needed no alteration. Nor is it felt that fire was used in shaping. But it is thought that fire often played an important role in making the mechanical processes less difficult during some stage of manufacture prior to final retouch.

Holmes (1919) compiled a summary volume entitled Aboriginal American Antiquities, the sources for which are nearly all pre-twentieth century as a perusal of the bibliography indicates. This book served as a springboard to the past since Chapter XXXV is entitled "Fire Fracture Process" and contains a number of useful references; those of which were available, in turn, led to others. Many of these early publications were not available. Despite the fact that complete coverage was not possible, it soon became apparent that man's use of fire in connection with his chipped stone tool-making technology fell into three main categories: (1) exposure of flint to fire as an aid in the chipping process, (2) use of fire in quarrying operations, (3) caches.

Exposure of Flint to Fire as an Aid in the Chipping Process

This category deals primarily with firsthand accounts or, perhaps more accurately, fanciful interpretations of firsthand accounts.

It seems appropriate to mention the following well-known description which appears to have had its genesis with Herman Lehmann:

> We threw a large flint stone, from two to six feet in circumference, into the fire. After the stone became very hot, small thin pieces would pop off; we selected those pieces which would require the least work to put into shape, and picked these pieces up with a stick split at the end; while these pieces were very hot, we dropped cold water on those places we wished to thin down; the cold water caused the spot touched to chip off, and in this way we made some of the keenest pointed and sharpest arrows that could be fashioned out of stone (Wallace and Hoebel 1952: 105).

Though widely accepted by laymen, the foregoing has been largely discredited by most professionals. To those who are sophisticated in the art of flintknapping, it does seem like a waste of time to use this method even if one could control the dripping of water sufficiently to predict the type of flake that would be removed; the work would proceed much faster by using a percussor or pressure tool.

Along this same line, Holmes (1919: 364) cites "A remarkable account of the use of fire in chipping flint implements . . . furnished by Thomas H. Fraser" who

> was informed by Chief Paul, the head of a remnant of the Mic-mac tribe, resident on the northern coast of Nova Scotia, that in his grandfather's time, flint arrow-heads were made by the systematic application of fire and water, and I still have in my possession an arrow-head made according to the process described by him.

Holmes (1919: 364-5) quotes accounts of the Digger Indians on the eastern side of the Sacramento River and of the Seri Indians of western Sonora, Mexico, who employed essentially the same technique. It would appear that these methods were not actually observed by the recorders.

Ellis (1940a: 42) furnishes other accounts pertaining to the Athabascan use of this method and describes several experiments where "chipping" in this way was attempted in order to settle the question as to whether dripping water to remove flakes is fact or myth.[1] He says:

> Experimental attempts to duplicate this fracturing technique have shown it to be very unsatisfactory. In the first place flint exposed to an open flame for even a short length of time will heat through and shatter into angular fragments and tiny flakes. The portions of the flint which do not shatter may be treated with cold water dropped from the end of a stick or by other means with little reaction. The usual result of the application of cold water to hot flint is the boiling and rapid evaporation of the drops of water. Occasionally small chips may fly off, but their direction and position cannot be controlled. Dropping thoroughly heated flint into a pan of cold water will simply remove tiny fragments which are broken away by the sudden change in temperature, but often even this treatment has negative results. Also the flint which has been subjected to fire is so filled with tiny fire cracks and the surfaces of the material so roughened due to the differential expansion of the crystals caused by the heating, that it is impossible to use it to any practical advantage in the shaping of stone implements. An examination of thousands of specimens in the museum failed to indicate that fire played any part in their manufacture (Ellis 1940a: 43).

Pond's (1930: 25) account is in complete agreement with Ellis.

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[1] An experiment was carried out which is described in the section on Methodology that was conducted prior to a knowledge of Ellis' work. The results were identical.

Schumacher reports that

> The rock is first exposed to fire, and, after a thorough heating, rapidly cooled off, when it flakes readily into sherds of different sizes under well directed blows at its cleavage (1877: 547).

Powers records the following:

> It was a source of wonder to me how the delicate arrow-heads used on war-arrows with their long thin points could be made without breaking them to pieces. The Viard [Wiyot] of northern California proceed in the following manner: Taking a piece of jasper, chert, obsidian, or common flint which breaks sharp-cornered and with a conchoidal fracture, they heat it in the fire and then cool it slowly, which splits it in flakes (1877: 104).

Other authors (e.g., Mason 1887: 226; Fowke 1896: 172) refer to the Powers and Schumacher accounts but add nothing pertinent. The methods described by Powers and Schumacher are more plausible than those avowing that arrowheads are made by dripping cold water on hot stones; however, it should be pointed out that they are in the minority.

The above references constitute the totality of the available information on this subject with regard to American Indian practices unless some undiscovered source exists.

The author is in frequent correspondence with Kenneth P. Oakley of the British Museum, London. He was not aware of this technique at all and could not "recall reading any account of any people heating stone before finishing in projectile-point form" (Oakley 1970, personal communication). Another European scholar, François Bordes

> . . . laughed until he tried heating some flint and then he became convinced. He has now traced the thermal treatment back to Solutrean [see Bordes 1968: 159] and both he and Tixier have done extensive work on experimental heat treating (Crabtree 1969, personal communication).

Thus, there appears to be even less information from European sources than from the New World. However, the following account concerning the Andaman Islanders demonstrates that the intentional heating of stone to cause alteration was not unknown to certain groups in the Old World. This is even the more interesting since the Andaman Islanders were reported to have no knowledge of how to kindle fire.

> Chips and flakes are never used more than once; in fact, several are generally employed in each operation. . . . Flaking is regarded as one of the duties of women, and is usually performed by them.
>
> For making chips two pieces of white quartz are needed; the stones are not pressed against the thigh, nor are they bound round tightly so as to increase the line of least resistance to the blow of the flaker; but one of the pieces is first heated and afterwards allowed to cool, it is then held firmly and struck at right angles with the other stone: by this means is obtained in a few moments a number of fragments suitable for the purposes above mentioned . . . (Man 1883: 380).

A paraphrasing of this quotation is to be found in Mason (1895: 137).

Use of Fire in Quarrying Operations

The information contained in this category comes from archaeological excavations and observations made at quarry sites no longer in operation.

The most widely quoted use of fire in aboriginal quarry sites is that of Fowke in his description of Flint Ridge, Ohio:

> . . . Traces of fire were plainly visible in the pits, from which the inference is natural that fires were built upon the rock, and that, while heated, water was thrown on it. The stone could thus be broken into pieces (Wilson 1897: 870).

Holmes (1919: 176-7) quotes Fowke in a similar description of the use of fire discovered in another pit at Flint Ridge, then adds

> In general, however, the action of fire is destructive to stone, and if not very discreetly employed will so flaw the stone as to make it unfit for most uses. Fowke tells us how this destructive tendency was probably avoided by the ancient quarrymen of Flint Ridge. According to his determination, fire was built upon the surface of the flint body, such portions of the purer stone as were desired for use being protected from the action of the heat by layers of moist clay (Holmes 1919: 364).

At the novaculite quarries near Hot Springs, Arkansas, which are possibly as extensive as those of Flint Ridge, Ohio, Holmes (1919: 198) observed some use of fire in the quarrying operations.

Ellis (1940a: 45) describes an experiment conducted by William G. Mills at Flint Ridge which may cast some doubt on the use of fire in quarrying operations:

> ". . . here was the bed of flint uncovered and an abundance of dry wood at hand. The fire was kindled, and was kept burning for two hours, producing an intense heat on the underlying surface of the flint. The fire was then removed and two buckets of cold water were thrown upon the surface. I fully expected the flint to break in large pieces, but it merely checked and cracked into small pieces to the depth of perhaps half an inch. After the conclusion of this experiment it was apparent that fire as a direct agent in quarrying of flint was perhaps not effective."

The following account of Indian jasper mines in the Lehigh Hills of Pennsylvania is interesting because it hints at the possible use of fire to fracture stone in a manner similar to the historic observances described in the previous category:

> Scattered fragments of charcoal were scarce in shaft 12 below the ninth foot, but all the other diggings and dumps were sprinkled thick with bits of

charcoal. About 20 per cent of the chips and 10 per cent of the large blocks were reddened as if by fire, while reddened fragments were abundant in all the fire-places. Nothing was surer than that fire had played a great part in the quarrying process; but while four fire-places examined showed no trace of cooking, they also gave no sure clue to their purpose, and there would have remained a doubt whether the fires had not been built for warmth had not a fifth hearth discovered in shaft 2, at a depth of 15 feet, seemed to settle the question. It was an oven regularly built of blocks of jasper and contained a mass of charcoal and ashes. The fact that the sides of the blocks were reddened, and several had already split through the middle, while the interstices were filled with fine splinters, offered conclusive evidence that the quarrymen had built the fire to fracture the blocks, which measured 2 feet, 1-1/2 feet, 6 and 7 inches respectively in diameter.

My experiments proved (a) that if a large block of two feet in diameter is thoroughly heated on a wood fire it breaks into numerous pieces at a moderate blow; (b) that only the fragments near the fire are reddened; (c) that the fragments lose their original gloss by the process. The luster, however, seemed to be regained by long burial in damp clay, as was indicated by the high-polished fracture of some of the reddened chips found on the fire-places. Moreover, many of the worked forms gathered on the surface had been probably fire-reddened, and it is not unlikely that the Indian could have so heated the blocks as to reach their purer parts without spoiling the whole, while many of the large and coarse blocks might have been fire-fractured to get them out of the way (Mercer 1894: 84-6).

Oakley (1969, personal communication) says "I have come across evidence at several localities suggesting that stone age craftsmen sometimes obtained flakes of intractable stone by using the method of 'fire setting,' . . ." Also

At Hangklip on the coast east of the Cape Peninsula, South Africa, erosion of peat has revealed a Late Acheulian factory site. At the base of large blocks of Table Mountain Sandstone protruding through the peat there are quantities of large thermal flakes, and on the surrounding ancient ground surface there are many broken or incomplete hand-axes and cleavers made from such flakes. Professor A. J. H. Goodwin has suggested that Acheulian man obtained the flakes by the method of 'fire-setting,' that is to say burning fires around the blocks of sandstone and then dashing

> water on to the heated rock to cause exfoliation. However, as far as I am aware, no ashes have been found around the blocks (Oakley 1955: 45).

This report is particularly interesting since it suggests that at a very early time man had a sophisticated knowledge not only of stone fracturing processes but also of the use of fire.

Caches

For many years stone caches consisting of arrowheads, spearheads, grooved axes, polished stone hatchets, large chipped flints, spades, etc., have been reported in the literature and there has been much speculation concerning why these were deposited. It seems perfectly reasonable to suggest that a flint cache as described recently by Hammatt (1970: 141) was used as a paleo-Indian butchering kit. Other suggestions that have been encountered in the literature are that the stones were (1) buried to keep them moist; (2) buried to hide them from enemies; (3) used as grave goods; (4) unfinished preforms to be completed later. It therefore is fair to add yet another suggestion: some "caches" existed because the buried flints were being thermally altered in order to make final chipping easier. While only a small number of descriptions mention the presence of charcoal, it seems plausible to assume that a farmer plowing a field who uncovers as many as several hundred flint implements at one fell swoop is not going to stop to notice that the pit also contains evidence of fire. Therefore, the number of references might be multiplied many times if the excavations had been conducted in the mid-twentieth century.

Montgomery County, New York: . . . a cache of 117 arrowpoints on the farm of . . . near a spring. They lay about 6 inches below the surface, on a bed of ashes 3 inches thick, which rested on a hearth or fireplace, about 10 feet square, of cobblestones from the drift. The arrowpoints average about 3 inches in length and are of dark-blue and gray flint, leaf-shaped (Wilson 1897: 971).

Old fort and village site in Saline County, Missouri: Three feet farther from the center was the edge of a pit 5 feet in depth and 6 feet in diameter. At one point on the bottom was a pile of minute flint chips scaled off in making implements of small size or delicate finish; there were enough of these to fill a pint cup. A slightly smaller quantity of similar chips lay higher up (Fowke 1910: 90).

Dutchess County, New York: While employed in digging, his spade brought up a number of arrow-points. He described them to be nicely piled side by side, edgewise, in two or three rows. There were perhaps two or three hundred in all. On each side and on top were some charred logs and sticks, that seemed to be the remains of an old fire. They were 10 or 15 inches below the surface of the pond. They are of a blue jaspery flint, and seem to be in an unfinished condition. I thought that probably the Indians had brought them from a distance (as I have never found any of the same rock anywhere in this neighborhood) and made this pocket and covered the traces of them by building a fire, intending to return and finish them at their leisure; or, perhaps, they hid them there to prevent their capture by their enemies (Shepard 1877: 307-8).

In 1894, Dr. J. F. Snyder excavated Mound No. 1 of the Baehr Group, on the west side of the Illinois River, thirteen miles below Beardstown, and opposite the mouth of Indian Creek [Illinois]: At the base of the mound was an oval of clay on which "was a mass of black hornstone implements, that apparently had been thrown down in lots of 6 to 20, with sand over and between each lot, as though to isolate them from each other. This deposit of 6,199 flints was covered with a stratum of clay, 10 inches in thickness; and on this a fire had been maintained for some time . . . . The flints forming the nucleus of this mound are very . . . rudely fashioned; some are quite neatly finished, but the greater part of them are only chipped and ill-shaped. . . ." . . . they do seem to be unused blanks (Ellis 1940b: 112).

It is interesting to note that most of these accounts mention charcoal and the unfinished condition of the finds.

This is exactly what one would expect if these "caches" consisted of preforms to be thermally altered prior to final chipping. There would be no reason why they couldn't be left for extended periods until needed. Perhaps they were never recovered by their owners because European introduction of iron and/or disruption of the aboriginal way of life intervened.

Discussion

In addition to the references cited there are other accounts tantalizingly suggestive of thermal alteration of rocks used to make projectile points. It is tempting to interpret these descriptions as evidence of heat treating simply because it would be rewarding to discover an accurate account; however, they must be considered conjectural until proved otherwise. This might be accomplished by reexamining cache collections as well as conducting excavations with this problem specifically in mind.

It is possible that pertinent publications may have been overlooked (many were not available), but it is unlikely that there exists anywhere in early documents the step by step procedure involved in thermally altering siliceous materials under controlled conditions. Several authors, most of whom have already been quoted, consistently mentioned the use of fire. A summary of this information might be as follows:

> These processes [of shaping] are distinguished by such terms as breaking, flaking, . . . All are purely mechanical; none are chemical, save a possible use of fire to induce changes in the rock in some parts of the quarry work (Holmes 1893: 25).

It may be concluded, therefore, that Crabtree made a significant and original contribution with his observation, experimentation, and description of this phenomenon.

> I first discovered the thermal treatment when I was about 17. I would find worked aboriginal material of superb quality, but when I found the raw material source it was never the same and had an entirely different texture. I finally wondered if they had in some way treated the stone and so tried the old coal range and heated the rocks which I buried in sand. After much trial and error, I was able to duplicate the texture of the worked pieces and decided this was what they must have done. I could get no one to accept the theory, however, until the Les Eyzies conference in 1964. Tixier accepted it readily as he had encountered the same complex problem in Algeria (Crabtree 1969, personal communication).

Since 1964, many archaeologists throughout the New and Old Worlds are beginning to suspect that much of the stone remains recovered from archaeological sites has been subjected to thermal treatment.

Since the Literature Review has revealed no accurate early description relative to this problem that might be applied to archaeological finds, at least a portion of the major contribution aimed at in this investigation seems to have failed, i.e., it should be possible to demonstrate that prehistoric peoples were aware of the advantages conferred by thermally altering their lithic materials. However, enough accounts exist describing the use of fire in some phase of the manufacture of flint tools that the evidence cannot be considered entirely negative. It is not surprising that accurate accounts do not exist when it is taken into consideration that:

1. Europeans who first encountered the American aborigines were concerned with matters other than stone technology, e.g., survival in a new ecological situation.

2. It was more fashionable to record esoteric ceremonial performances than the practical aspects of native life.

3. Iron was almost immediately substituted for stone in making implements; in fact even prehistorically in the Southeastern United States, arrows and spears were often headed by materials other than stone: bird bones, bird bills, fish scales, fish teeth, fish fins, animal bones, animal teeth, horseshoe crab tails, and oyster shells. Some had no separate head--the wood or cane of the arrow shaft was merely sharpened and served as the point.

4. Since chipping stone tools (except for gun flints) was not part of European technological knowledge at the time of contact, not enough was known to observe or describe the procedure accurately, much less interpret such an alien process.

5. Most explorers, adventurers, etc., were not among the Indians for extended periods. It is possible that this procedure occurred only at special times or in specific locations and was considered too mundane to mention.

To establish that desirable changes do occur when siliceous materials are subjected to critical temperatures, it was necessary, therefore, to turn to nonanthropological literature. These publications will be cited in appropriate places in the section on Methodology.

MATERIALS

The terms _chert_ and _flint_ have been used interchangeably and it is difficult to determine from the literature if any actual structural differences exist between them.

> _Flint_ is a term widely used both as a synonym for chert and for a subvariety of that material. Tarr says that flint is identical with chert in texture and composition and the term, therefore, should be dropped or reserved for artifacts (Pettijohn 1949: 320).

Thus, in describing chipped stone tools, one might say, for example, that flint artifacts were manufactured of Arkansas novaculite, Pennsylvania jasper, etc. This is, of course, already being done to a certain extent and to those interested in lithic technology information of this nature is important since it contributes greatly to an understanding of the kind of workmanship that might be expected or whether trade relationships existed if the material is not local.

Most archaeologists would define flint as a rock composed of microcrystalline quartz that breaks with a conchoidal fracture. This is an adequate general description, but does not suffice when specific materials from diverse geographic areas are being considered. From the standpoint of this investigation, knowledge of the formation and composition of siliceous materials native to the state of Florida is necessary since alteration by heat might not take place at the same temperature in materials whose structure is slightly different.

Chert, being a rock, varies greatly in its physical characteristics even though the basic components, the minute crystals of quartz, are uniform in character. The factors that determine the physical properties of cherts are: (1) the size of the quartz crystals; (2) how the anhedral crystals fit together affecting porosity and fracture; (3) the amount of foreign material present, fossil replacements, and other heterogeneities including flux compounds; (4) void spaces; and (5) crystalline fabric (the crystals are not equidimensional or always randomly oriented). Thus, in the Florida cherts, there are different types and even a single nodule is not necessarily homogeneous throughout its mass.

The lithic raw materials used almost exclusively in this study were Florida cherts. Justifications for using a general term such as <u>chert</u> are as follows. To this author, at least, the term <u>flint</u> indicates an extremely fine-grained material found in the chalks of England. Also, as stated above, flint is used as a common designator for artifacts with no specific material intended. Using the modifier <u>Florida</u> preceding the word <u>chert</u> calls to mind a particular type of material much as Arkansas novaculite, Pennsylvania jasper, or English flint does. Though there is a range of differing textures and homogeneities, Florida cherts were all formed under similar conditions and share common characteristics which distinguish them from other siliceous rocks.

There are a number of conditions under which chert will form. Florida cherts occur as a secondary formation due

to the replacement of carbonates with silicas. All of the chert deposits in Florida are in relationship to relict clay hills in contact with limestone; these generally correspond to the Ocala Arch where siliceous deposits lie unconformably over the Ocala Limestone. Cherts also occur at the edge of the arch where limestones are interbedded with siliceous deposits. The occurrence of cherts is thus in the upper part of the Eocene Ocala Limestone, and the Oligocene and Lower Miocene Formations bordering the areas of the Ocala Uplift. Cherts found on the Ocala Arch have replaced limestones that are Eocene in age and those such as occur in Hernando and Hillsboro Counties have replaced limestones that are Oligocene and Lower Miocene in age. Cherts in the Miocene deposits are found as far south as Zulpho Springs, Hardee County. There are no cherts naturally outcropping along the east coast of Florida or in south Florida (Brooks, personal communication). Figure 1 shows the extent of the areal distribution of formations that might contain chert in peninsular Florida.

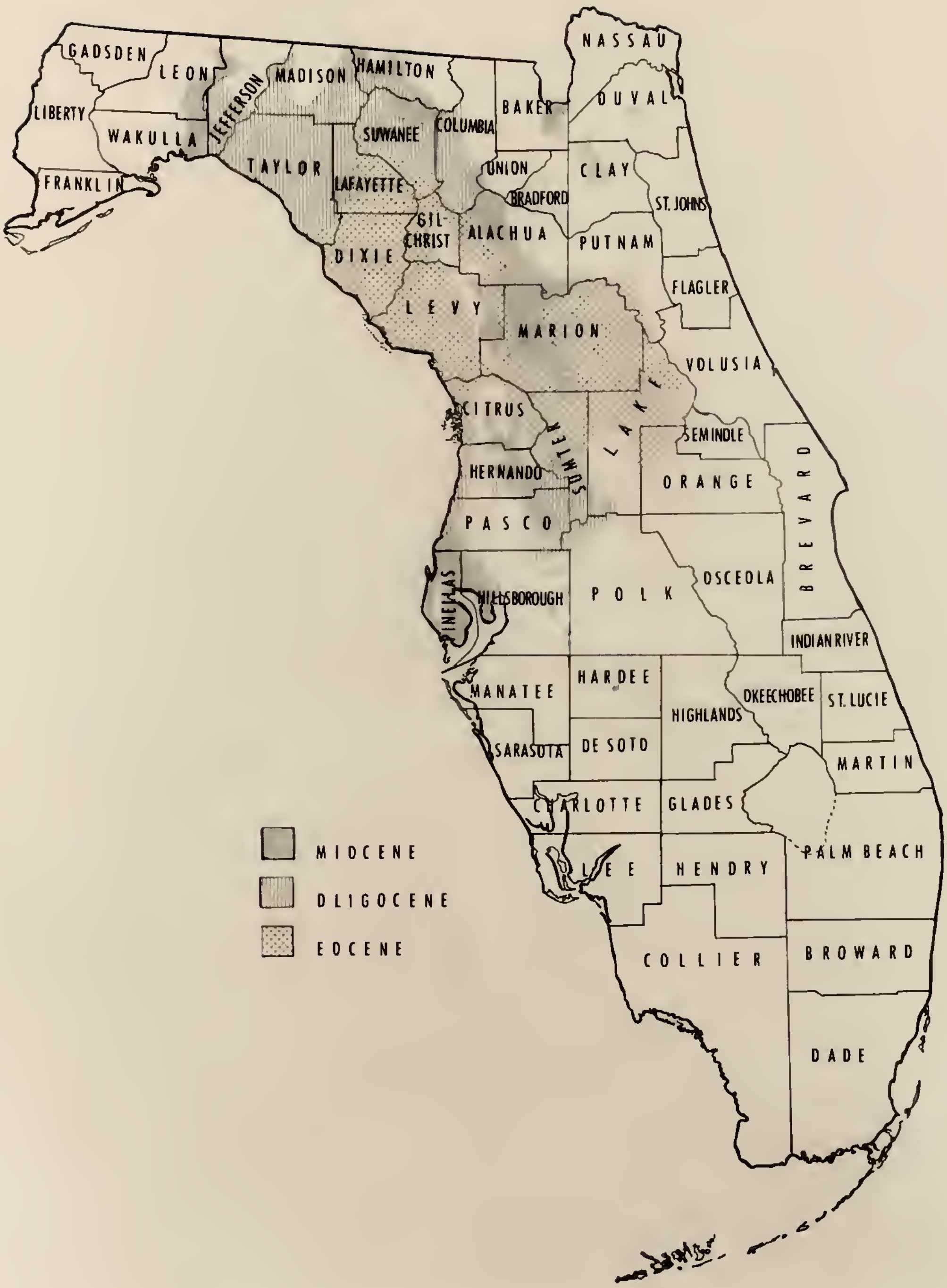


Figure 1.--Areal Distribution of Outcrops Probably Containing Chert in Peninsular Florida (adapted from Cook 1945, Plate 1; Brooks, unpublished geologic map of Florida).

METHODOLOGY

The experiments described in this section were conducted primarily to determine whether some sort of alteration takes place in microcrystalline rock types upon heating that makes them easier to flake, especially to pressure flake. If such a change does occur, the author proposed to determine at what temperature alteration takes place and what are the physical or chemical changes that cause alteration.

Whenever comparisons are made between heated samples and unheated controls, the specimens were obtained from the same core. An attempt was made to use material as free from fossil inclusions or other heterogeneities as possible and to obtain the samples from approximately the same area of the core since textural differences do occur; for instance, the area directly under the cortex is nearly always finer grained than further within the mass. Throughout this section reference is made to

1. Critical temperatures: this should be interpreted as 350°C-400°C for Florida cherts.

2. Slow rise in temperature: the materials being heated were subjected to 50°C elevations in temperature until the testing temperature was reached; they were left at each succeeding increment for a sustained period--usually 24 hours.

3. Rapid rise in temperature: the materials being heated were subjected to 50°C elevations in temperature until the testing temperature was reached; they were left at each succeeding increment for a short period--generally 1 hour.

Heading Experiments

All of the experiments involved the use of heated and unheated specimens but it was necessary to create a special category describing the many and varied conditions under which the rock materials were thermally treated. These tests followed what might be considered an evolutionary sequence since the results of one experiment often dictated the need for another which in turn suggested still others. As pointed out in the Literature Review, only Crabtree and Butler's (1964) article discusses heating of siliceous materials from an archaeologically significant point of view; therefore, these experiments are of a pioneering nature.

Weight Loss

Twelve 1-inch cubes, each weighing approximately 42 grams, were laboriously prepared to be used for compressive strength tests. (See heading entitled Strength Tests for the description and results of this experiment.) Six of these samples were treated as follows. Two 1-inch cubes each of obsidian, silicified coral, and Ocala chert were weighed prior to subjecting them to heat. They were placed in a Blue M Lab-Heat Muffle Furnace, heated to 100°C for 48 hours, removed, placed in a desiccator, and weighed after they were thoroughly cooled. They were then reheated to 100°C and left an additional 48 hours to determine if all the moisture that would be driven off at 100°C had been removed during the original 48-hour period. It had been removed, as was indicated by no change in weight. This same

procedure was followed for 150°C, 200°C, and so on through 500°C but it was not felt necessary to subject the specimens to such long periods of heat or to reheat them at each succeeding temperature since no additional significant weight loss occurred with prolonged heating or subsequent heating at the same temperature. (The validity of this procedure is established when the next experiment is described.) The results of this experiment are given in Table 1.

Twenty-six specimens consisting of samples from 14 chert nodules obtained from various areas of Florida plus one specimen of English flint were weighed, heated, reweighed, reheated, and reweighed again through successive temperatures to 500°C in the following way. After the initial weighing, the samples were heated at 100°C for 54 hours after which they were removed from the oven while hot, placed immediately in a desiccator, and reweighed when thoroughly cool. They were returned to the oven and again heated to 100°C for an additional 4 hours and subjected to the same procedure as described above. This process was repeated until 500°C was reached. The oven was taken to the testing temperature at 50°C increments, left at each increment for 1 hour, and then moved up. In other words, it took an additional hour to reach each succeeding temperature. The only variation which occurred within the experiment was that it was not considered necessary to subject the material to a total of 58 hours at each temperature as had been done at 100°C. The fact that no significant additional weight loss occurred by reheating at the same temperature justifies

TABLE 1

HEATING EXPERIMENT TO DETERMINE WEIGHT LOSS OF 1-INCH CUBES

Sample	Percentage Weight Loss									
	96 hr[a]	144 hr[a]	48 hr	48 hr	48 hr	24 hr	24 hr	24 hr	24 hr	Total
Obsidian	0	0	0	0	0	0	0	0	0	0
Obsidian	0	0	0	0	0	0	0	0	0	0
Silicified coral	.36	.33	.04	.05	.11	.06	.15	.23	.07	1.40
Silicified coral	.49	.33	.04	.06	.06	.14	.19	.19	.08	1.58
Ocala chert	.32	.06	.01	.03	.04	.06	.17	.13	.07	.88
Ocala chert	.32	.05	.01	.02	.07	.09	.07	.15	.09	.87

[a]The total hours of heating for the 100°C and the 150°C increments actually were split (that is, 48 and 48, 72 and 72) to determine if additional weight loss occurred when reheated at the same temperature. When it was determined that no additional weight loss occurred these two times were combined and the procedure was discontinued.

the reduction in heating time. It should be pointed out that most of the samples tested did not exceed 20 grams. Had they been larger, longer heating periods might have been necessary. One sample, however, weighed 95 grams and the percentage weight loss for this sample was comparable to that of smaller samples of the same material. See Table 2 and Figure 2 for the results of this experiment. Figure 3 graphically illustrates typical weight loss patterns for Florida cherts.

A number of samples which had been used for the experiment described above together with their unheated controls were placed in moist pure quartz sand and left for one month. Water was added to the sand periodically to maintain dampness. Heated and unheated samples of the same material were kept as controls at room temperature. The purpose of this experiment was to determine how much moisture the already heated materials would absorb as compared to unheated samples subjected to the same conditions. The amount of moisture taken up by the heated samples was greater than that of the unheated. The heated and unheated controls left at room temperature during the month yielded some interesting information. The controls, that had already been subjected to heat, lost less weight than those which had not been heated. See Table 3 for the results of this experiment. The soaked samples were then subjected to heat to determine if there was any significant difference in the amount of weight loss between the heated and unheated samples or in the temperature at which the weight loss occurred. See

TABLE 2

HEATING EXPERIMENT TO DETERMINE WEIGHT LOSS OF SAMPLES FROM DIFFERENT LOCATIONS[a]

Sample	Percentage Weight Loss									
	100°C 58 hr	150°C 40 hr	200°C 28 hr	250°C 22 hr	300°C 16 hr	350°C 8 hr[b]	400°C 9 hr	450°C 5 hr	500°C 4 hr	Total
Ah1	1.08	.14	.05	.03	.05	.11	.15	.10	.15	1.86
Ah2	.67	.19	.11	.05	.03	.07	.13	.11	.11	1.47
Bh	.67	.04	.02	.02	.02	.14	.14	.16	.12	1.33
Ch1	.38	.05	.02	.03	.02	.09	.11	.10	.11	.91
Ch2	.45	.02	.02	.02	.03	.10	.07	.13	.10	.94
Dh	.57	.03	.01	.00	.03	.08	.13	.10	.10	1.05
Eh1	.49	.01	.01	.00	.01	.06	.12	.09	.10	.89
Eh2	.38	.03	.02	.01	.00	.07	.10	.08	.06	.75
Eh3	.34	.02	.02	.03	.02	.03	.10	.09	.10	.75
Fh1	.51	.04	.04	.02	.01	.07	.16	.15	.12	1.12
Fh2	.42	.07	.05	.00	.02	.08	.15	.09	.13	1.01
Fh3	.49	.02	.03	.01	.03	.05	--c	.13	.10	--
Gh	.86	.03	.04	.03	.06	.10	.16	.12	.10	1.50
Hh1	.92	.00	.04	.02	.00	.12	.15	.19	.18	1.62
Hh2	.63	.06	.03	.00	.01	.08	.12	.15	.14	1.22
Ih	.60	.09	.04	.06	.10	.14	.12	.19	.08	1.42
Kh1	.64	.03	.01	.01	.01	.04	.17	.13	.13	1.17
Kh2	.64	.04	.02	.01	.03	.08	.18	.16	.12	1.28
Mh	.77	.08	.07	.05	.00	.09	.17	.16	.10	1.49
Nh1	.49	.03	.03	.02	.02	.08	.13	.09	.15	1.04
Nh2	.35	.02	.03	.02	.01	.06	.13	.08	.14	.84
Nh3	.40	.04	.02	.03	.00	.09	.14	.13	.12	.97
Nh4	.44	.03	.03	.02	.03	.07	.15	.08	.11	.96
Ph	.71	.15	.07	.03	.11	.11	.15	.13	.12	1.58
Sh	.14	.04	.03	.01	.02	.00	.00	.00	.00	.24
Th1	.88	.08	.05	.01	.02	.09	.15	.18	.11	1.57
Th2	.85	.05	.06	.01	.02	.10	.17	.17	.15	1.58

[a]Samples A and P are silicified coral; samples B, C, and E are cherts from High Springs, Florida; S is English flint; all other samples were obtained from differing locations such as Johnson Lake in Levy County, three miles north of Ocala in Marion County, and from Alachua County. All of these samples were heated twice at the same temperature but the results were combined because the second heating produced no significant additional weight loss. It should be pointed out that if the total period of heating at 450°C had been for a longer duration perhaps there would have been less weight loss at 500°C and thus would have resembled more nearly the results of Table 1.

[b]These samples were heated for a total of 12 hours but the results of the second test of 4 hours were not reliable.

[c]This sample fractured at 400°C.

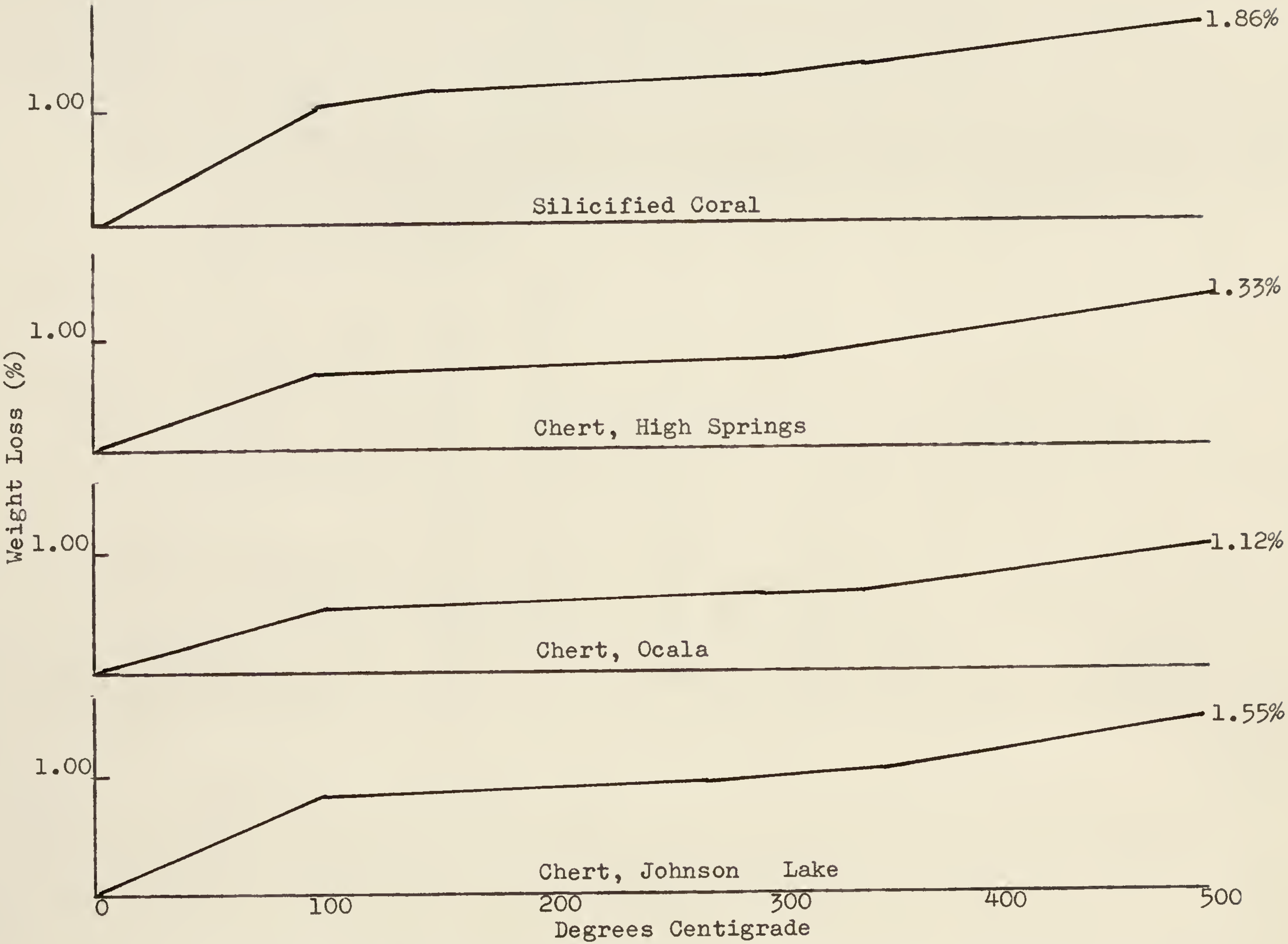


Figure 2.--Weight Loss upon Heating of Cherts from Various Locations in Florida (note the rapid increase beginning around 350°C).

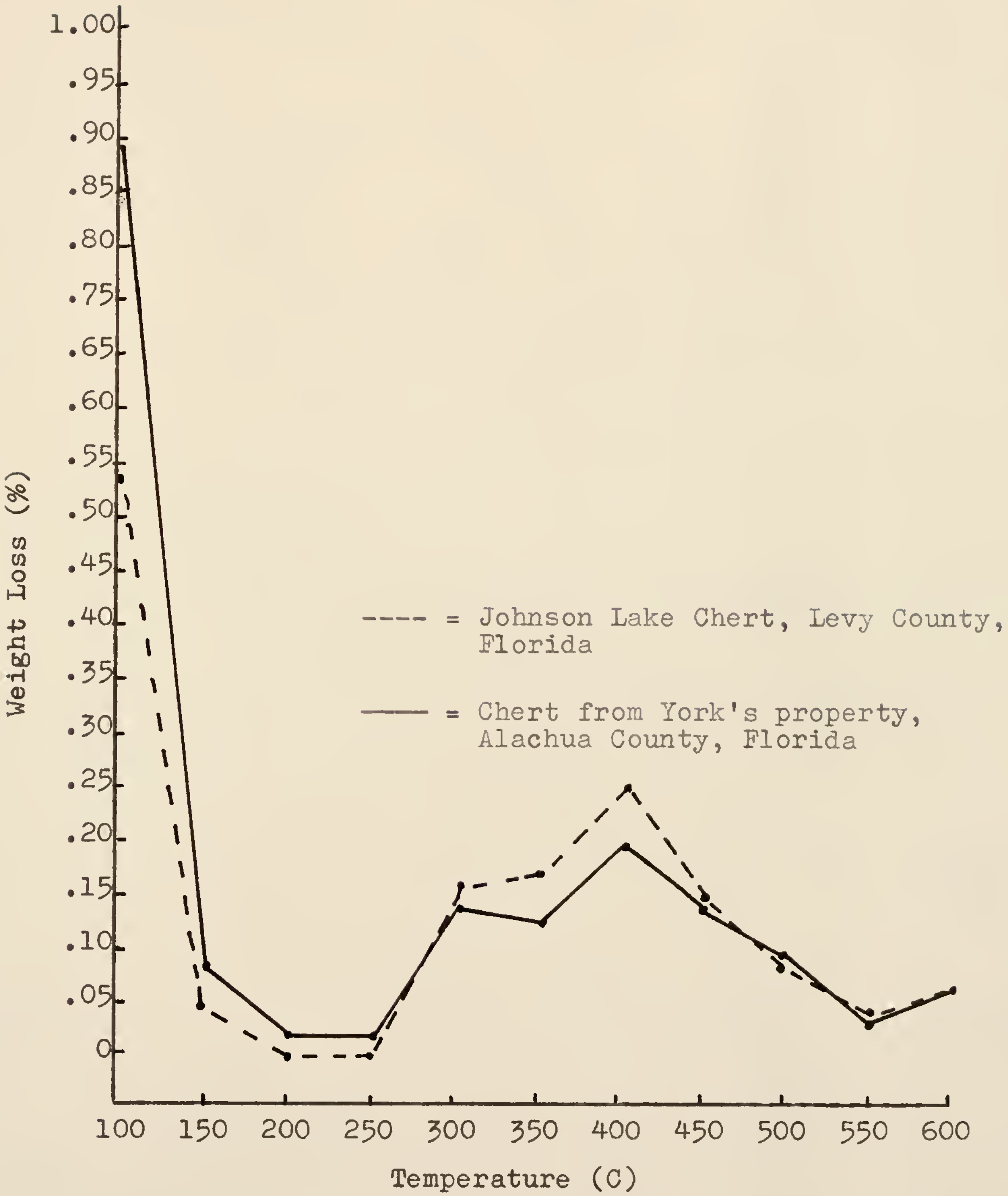


Figure 3.--Weight Loss of Samples of Florida Chert Illustrating the Typical Pattern Observed for All Florida Specimens Tested Throughout a Two-Year Period (Obsidian, Arkansas Novaculite, and English Flint did not follow this pattern).

TABLE 3

RESULTS OF EXPERIMENT CONDUCTED TO DETERMINE ABILITY OF HEATED ROCK SAMPLES TO TAKE ON MOISTURE

Sample[a]	Weight Gain after One Month in Moist Sand Bath		Sample[a]	Weight Loss of Control after One Month at Room Temperature (%)
	24 hr after Removal (%)	4 Days after Removal (%)		
Ah	.57	.53	Ah	.01
Ah	.45	.42	A	.06
As	.27(lost)[b]	.39(lost)	Eh	.00
Eh	.95	.27	E	.05
Eh	1.13	.35	Fh	.00
Es	.55	.09	F	.08
Fh	.18	.15	Hh	.08
Fh	.09	.03	H	.08
Fs	.04	.00	Nh	.01
Hh	.56	.44	N	.06
Hh	.72	.30	Th	.00
Hs	.42	.21	T	.00
Nh	.27	.21		
Nh	.26	.18		
Ns	.12	.01		
Th	.44	.36		
Th	.44	.40		
Ts	.28	.18		

[a] _h_ following the sample letter indicates that it has been heated; _s_ following the sample indicates that it was soaked but not heated; a letter with no _h_ or _s_ following it is an unheated and unsoaked control. The tremendous difference in weight after four days in the _E_ series may be because it is more calcareous.

[b] It is difficult to account for this anomaly unless an error was made in the original weight.

Table 4 for the results of this experiment. It was hoped that this experiment, though simple, might prove to be a reliable and inexpensive method to be employed by archaeologists to determine if chipped stone remains had been thermally altered. This will be discussed further under the heading Archaeological Application, but although there is a fairly consistent difference in weight lost on an intrasample level, on an intersample level there is overlap. Therefore, even if an investigator knew a great deal about the rock he was recovering (that is, source, composition, etc.), he would have to be careful in assigning too much importance to weight loss. This experiment also indicates that the weight loss at 100°C is probably not too indicative since it would fluctuate greatly depending on how damp conditions were prior to heating. It has been demonstrated that an appreciable change in weight occurs if the samples are left at room temperature after being removed from a moist environment (see Table 3).

Materials from the field which were thought to have been thermally altered since they were quite lustrous and many exhibited a pinkish cast were heated in order to compare weight losses with other specimens that were known not to have been previously heated. The results are given in Table 5. If this table is compared with Table 2, it is apparent that there is no significant difference in the amount of moisture given off between the materials that had been suspected of being previously heated and those which were being heated for the first time. The results of this

TABLE 4

HEATING EXPERIMENT CONDUCTED TO DETERMINE DIFFERENCES IN HEATED AND UNHEATED SPECIMENS AFTER SOAKING FOR ONE MONTH

Sample	Wt Loss after 24 hr at 100°C (%)[a]	(%)[b]	Additional Wt Loss after 24 hr at 350°C (%)[c]
Ah	.42	.38	.18
Ah	.40	.37	.12
As	.91	.79	.32
Eh	1.08	.40	.03
Eh	1.29	.51	.03
Es	.89	.43	.16
Fh	.50	.47	.03
Fh	.22	.16	.17
Fs	.61	.57	.27
Hh	.86	.74	.08
Hh	1.07	.65	.35
Hs	1.00	.79	.25
Nh	.53	.47	.05
Nh	.57	.49	.04
Ns	.65	.54	.23
Th	.55	.47	.08
Th	.49	.45	.10
Ts	.87	.77	.35

[a]Calculated from weight of samples 24 hours after removal from moist sand bath.

[b]Calculated from weight of samples four days after removal from moist sand bath.

[c]While there appears to be a slight, yet unreliable tendency for the weight loss to be greater at 100°C for the unheated soaked samples, the greater weight loss is quite consistent for the unheated soaked samples between 100°C through 350°C. Because there is overlap in percentages between heated and unheated depending on the sample involved, this method, while consistent within samples,could not be used as an archaeological application. Even if the investigator had a profound knowledge of the materials with which he was dealing the results would be tenuous.

TABLE 5

HEATING EXPERIMENT CONDUCTED TO DETERMINE WEIGHT LOSS OF ARCHAEOLOGICAL SPECIMENS SUSPECTED OF HAVING BEEN THERMALLY ALTERED

Sample	Weight Loss		
	After 24 hr at 100°C (%)	After 24 hr at 400°C (%)	Total (%)
M	.19	.68	.87
N	.28	.76	1.05
O	.27	.78	1.05
P	.39	.41	.81
R	.45	.56	1.01
S	.35	.85	1.20
T	.47	.52	.99
U	.36	.55	.91
V	.44	.69	1.13
W	.22	.38	.60
X	.35	.52	.87
Y	.27	.40	.67
Z	.25	.48	.73
35	.42	.59	1.01

comparison plus the data derived from soaking samples for a month in a moist sand bath indicate that it is not possible to use weight gain or loss as a reliable criterion in determining if archaeological specimens had been subjected to heat.

Decrepitation and Explosion

It was no mystery to primitive man that fire could be destructive to rock (see Literature Review), but this investigation has attempted to demonstrate that when siliceous rock materials are cautiously subjected to heat, an alteration occurs which conferred an advantage to prehistoric man in the manufacture of his chipped stone implements. Man had many thousands of years to discover this fact and to perfect the technique. Since the use of fire and the use of stone were two of the earliest and most important items in the inventory of a physically and culturally evolving creature, it should come as no surprise that he gradually acquired an intimate knowledge of their attributes. The observations to be discussed below are probably much like those made long ago.

The materials involved in these experiments were all Florida cherts. Reactions might occur at different temperatures, or not at all, if other rock types were used. Sufficient samples of non-Florida materials were not available for experimentation. These should be tested eventually because the results would certainly be enlightening.

Webster's New World Dictionary of the American Language (1970) defines decrepitate: "to roast or calcine

(salts, minerals, etc.) until a crackling sound is caused or until this sound stops; to crackle when exposed to heat." Explosion occurs when the stress that is causing decrepitation exceeds the elastic limits of the material.

When this study was initiated, several large chert flakes were prepared in order to test their reaction when heated. The author had intended to raise the temperature slowly according to Crabtree's (personal communication) instructions but the oven was progressing in degrees centigrade while the author, unfortunately, was thinking in degrees fahrenheit. The oven consequently heated more rapidly than anticipated. At 400°C the rocks exploded. The results are shown in Figure 4. Subsequent experiments, conducted to determine the reasons for the rock failure, resulted in one of the major contributions of this study.

Figure 4 illustrates quite clearly several facts that refute objections which may be raised with regard to the intentional heating and subsequent flaking of lithic materials. The picture shows potlid fracturing and blocky, angular flakes with no bulbs of percussion. This kind of debris does not occur by intentional flaking. It differs markedly from the thinning flakes found on archaeological sites that are suspected of being thermally altered. It resembles exactly what one would expect to result from too rapid expansion and contraction as might occur in a forest fire or if a rock had been placed in or near a hearth. Subsequent testing of Florida cherts has revealed that:

Figure 4.--Explosion Resulting from Too Rapid Exposure to Heat (note "potlids" and blocky angular flakes).

1. No spalling occurred (except on rare occasions) when the temperatures were raised very slowly.

2. No explosion occurred even when the temperature was raised rapidly to 350°C, allowed to remain at 350°C for 24 hours, and then moved to 400°C.

3. Explosion occurred on all occasions at 400°C when the material was taken to 400°C without allowing the temperature to be raised slowly or at least leaving it at 350°C for an extended period. Smaller specimens did not explode as readily.

4. If the temperature was raised rapidly, explosion, or at least some spalling, occurred whenever the material was removed from the oven at 400°C without allowing it to cool first.

5. If the temperature was raised rapidly, explosion, or at least some spalling, occurred occasionally but not often when the material was removed from the oven at 350°C without allowing it to cool first.

6. Explosion did not occur at 300°C even when the material was removed from the 300°C oven immediately.

7. Interestingly, explosion rarely occurred at any temperature when the material was removed immediately from the oven if the temperature had been raised slowly and maintained at the testing temperature for a sustained period.

8. Explosion never occurred when the material was tested a second time at the same temperature.

9. When the temperature was raised rapidly a crackling noise (decrepitation) was often heard at 350°C and always heard at 400°C when the material was removed from the oven without allowing it to first cool. It was not heard at 300°C nor was it heard when materials were heated for a second time.

10. When samples were placed directly into a preheated oven at 350°C no reaction occurred after 1/2 hour but all except a sample of Ocala chert snapped in half when removed from the hot oven and exposed to air temperature.

11. When samples were placed directly into a preheated oven at 400°C, explosion commenced after approximately 20 minutes. The oven was turned off immediately; explosion continued intermittently until the oven cooled to about 375°C. All samples had exploded, including Ocala chert, with the exception of a sample of High Springs chert which did not snap even after removal from the hot oven.

Two samples that had been heated to 350°C for 24 hours were removed from the oven with tongs while hot (still 350°C); the tongs were warmed on the side of the oven before touching the hot stone. One of these samples snapped in half with a very loud sound (room temperature was about 75°C). Cold water (tap water) was dripped along the edge of the samples with a dental syringe to see if they would flake--they did not! (See Literature Review for accounts describing this alleged phenomenon.) The samples were then placed directly under the tap and cold water allowed to flow over the entire sample. This resulted in an audible hissing sound and a crazing of the material. The same procedure was followed with two samples that had been left at 400°C for an additional 6 hours. Crazing occurred and subsequent attempts to flake the material caused it to crumble. It was impossible to pressure flake this material because the flakes could not be removed in a predictable way. The specimen literally fell apart (see Figure 5).

> Failure on quenching was shown by irregular cracks differing in appearance from the smooth conchoidal fractures . . . (Pressler and Shearer 1926: 308).

Other experiments were conducted to test the significance of the crackling sound which occurred when specimens were removed immediately from a hot oven. These experiments will be discussed at the end of this section and in those dealing with Petrographic Analysis and Strength Tests.

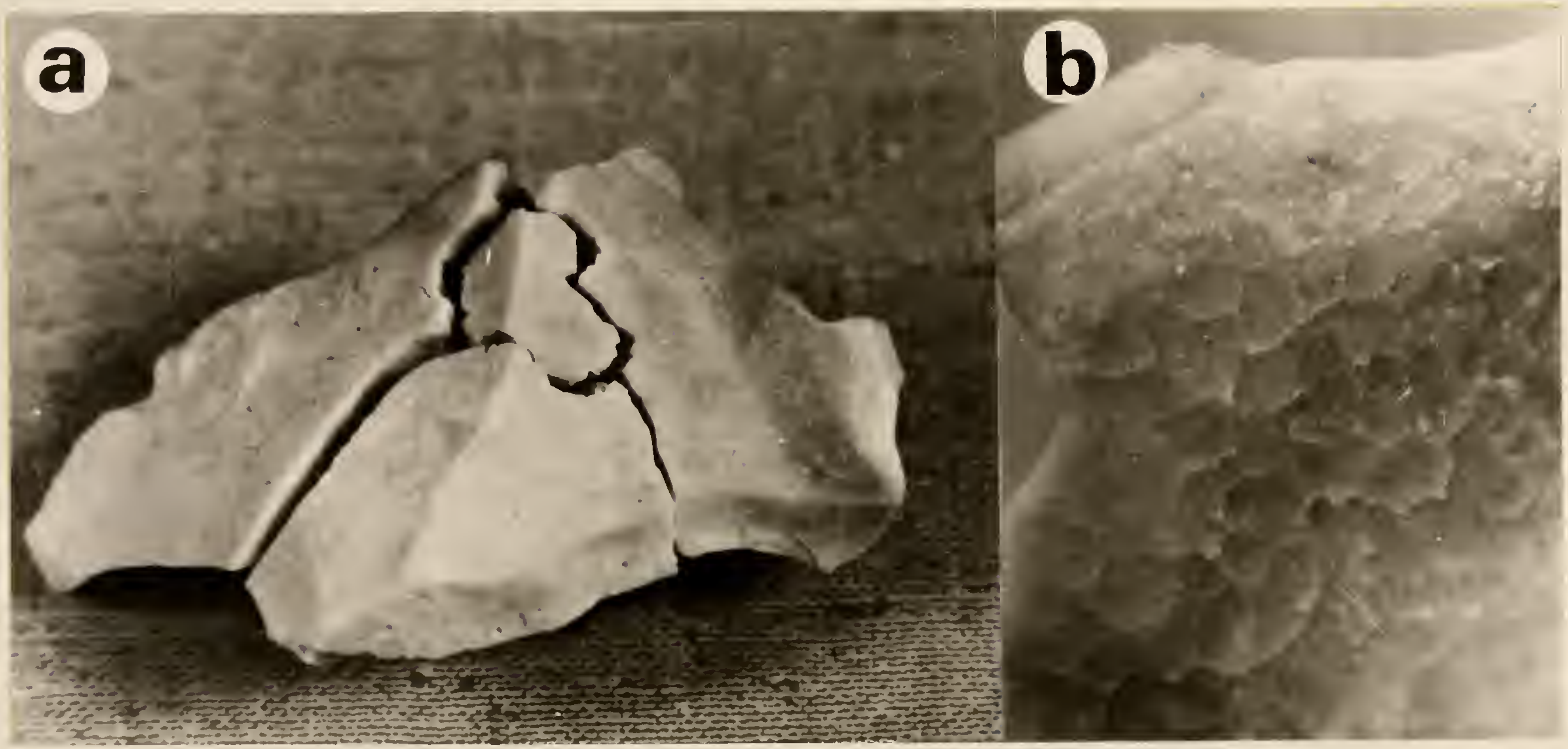


Figure 5.--Results of Experiment Conducted to Test the Validity of "Flaking" Hot Stones by Dripping Cold Water on Them: (a) The Rock Literally Fell Apart when Flaking Was Attempted (note deviation from conchoidal fracture typical of flint materials); (b) Magnified Area Illustrating the Crazing which Occurred.

Vitreousness

To vitrify is to convert into, or cause to resemble, glass or a glassy substance by heat and fusion. The more vitreous an object is, the more it has the luster of broken glass. In order for a cryptocrystalline rock to be converted into an amorphous, noncrystalline structure, it is necessary to subject it to temperatures of ca 1400-1700°C (approximately 3000°F). Aboriginal peoples in the state of Florida could not achieve or maintain temperatures this high. This fact is known by the quality of the pottery remains which indicates that pottery was fired at a much lower temperature--probably not over 550°C (ca 1000°F). Since the practice of altering lithic materials seems to have occurred on preceramic levels in Florida, it could not be assumed that earlier inhabitants were capable of producing temperatures higher than their descendants. Besides, from petrographic analyses, which will be described later, it has been demonstrated that the size of the cryptocrystals does not change even though vitreous luster occurs after heating.

Florida cherts are characteristically nonlustrous and coarse grained except for an area directly under the cortex which may be as thin as 1/8 inch or as thick as several inches, this latter being exceptional. Occasionally, however, the chert is slablike and ledgy, not exceeding an inch or two in thickness. When chert like this is found, the fine grained area generally extends from cortex to

cortex throughout the entire thickness of the rock and is extremely glass-like and homogeneous. Typically, however, the chert is found in thick rounded nodules or beds and the fine grained area under the cortex rapidly shades into a coarser grained area farther within the mass. The reason for this difference in texture is not clear but it occurs during replacement of the limestone by silica and might be "due to a descreasing rate of precipitation because of the diminishing rate of supply of solution as consolidation proceeds" (Folk and Weaver 1952: 500-1).

The smaller the size of the cryptocrystals, the more the material will exhibit the vitreous luster of glass on a fractured surface, and by contrast, the larger the cryptocrystals, the less lustrous is the aggregate mass in appearance. Interestingly, after coarse grained cherts are heated slowly to around 350°C, left for a period of time, and subsequently fractured, the fractured surface exhibits a glass-like luster but the grain size has not been changed. The reasons for this are quite simple and will be explained in the discussion which follows. Table 6 summarizes experiments using large and small samples of cherts from different rock masses and locations. These tests were conducted to determine the length of time necessary to effect alteration which results in a vitreous fractured surface. Each group of samples heated at each temperature was taken to 100°C and left for 2 hours after which they were raised by 50°C increments for 1-hour periods

TABLE 6

HEAT SOAKING EXPERIMENT CONDUCTED TO ASCERTAIN LENGTH OF TIME NECESSARY TO EFFECT THERMAL ALTERATION[a]

Sample No.	Hours at 350°C						Hours at 400°C					
	2	4	6	8	10	12	2	4	6	8	10	12
40			+	++	++	++	+	+	++	+++	+++	+++
50	+	+	++	++	++	+++	+	++	+++	+++	+++	+++
60	++	++	+++	+++	+++	+++	+++	+++	+++	+++	+++	+++

[a]If the space is left blank no change in vitreousness or flaking ease has occurred; + means change is slight; ++ change is pronounced; +++ change is complete. There was only occasionally noted a difference with regard to change and sample size. The smaller samples weighed approximately 3-8 grams; the larger samples weighed 50-80 grams. More attention was paid to thickness in selecting the sample than weight. Extreme crepitation occurred in the samples heated this rapidly to 400°C and in this regard, the larger samples were more involved than the smaller samples.

to either 350°C or 400°C as indicated, and then left at the ultimate temperature for the designated period of time. The oven was shut off and the samples allowed to cool in the oven. The gradual onset of vitrification seems to go hand in hand with an increasing ease in removing flakes. If alterations take place too rapidly, dramatic, destructive events occur. It should be emphasized that vitreousness is not apparent unless the rock specimen is broken or chipped after heating. The exterior surface of the heated stone remains dull. This is illustrated in Figure 6b.

Discussion

The information presented to this point has been mainly observation and description. The data now remain to be interpreted and the significance of the experiments discussed.

It seems fairly clear that no reliable inferences can be made from differences in weight loss and gain to help in determining if archaeological specimens have been thermally altered. Even under controlled conditions, though significant differences were observed for the same rock when flakes were either subjected to heat or kept as controls, nothing reliable was recorded when making comparisons between different rocks even when the method of exposure was identical.

In addition, the amount of water driven off, whether great or small, made no difference in the change which ultimately resulted as long as the temperature effecting

Figure 6.--Vitreousness Occurring when Florida Cherts Are Heated and Subsequently Flaked. Top: Specimens were Heated Slowly to 350°C, Left for 24 Hours, and Cooled in the Oven. Bottom: Specimen was Heated Slowly to 500°C, Left for 5 Hours, and Removed Immediately from the Hot Oven (note relict dull area that has not been flaked after heating).

the change was reached and maintained until the change occurred. Obsidian (Table 1) lost no weight at all but other tests suggest that a change might occur (see section on Strength Tests). A quartz crystal and pulverized quartz lost very little weight (after 24 hours at 350°C they had lost .01% and .04%, respectively), but it has been stated that quartz becomes easier to flake (Crabtree, personal communication; Man 1883: 380). English flint lost only about .15% to .25% and, because it is fine grained, we suspected that fine grained Florida cherts would lose little weight and coarse grained Florida cherts would lose more. However, there does not seem to be any significant, predictable difference between the amount of weight lost and crystal size in Florida cherts. This could be due to heterogeneities since even fine grained materials may have large void spaces that cannot easily be detected. It is more likely because, even though there is less water in any given interstitial space when the crystals are small, they are still anhedral and probably are not packed any better than larger ones; therefore, the same amount of water exists even though the crystals which it surrounds differ in size. The difference in weight loss for English flint compared to Florida chert might be due to the conditions under which they were formed. Florida cherts probably contain more water vacuoles. The cryptocrystals of English flint are probably more closely packed. The smaller the size of the crystals, the greater the surface area unless the crystals are intergrown.

If opal were present it might be a simple matter to explain what occurs when water is driven off through heating: the opal when dried would crack and adhere more firmly to the microcrystals exactly as jello would tend to stick to the side of a glass. This might result in a binding of the microcrystals. The presence of opal as interstitial material in chert, however, has been quite thoroughly dismissed (Folk and Weaver 1952; Schmalz 1960). Attempts to detect the presence of opal in this study produced only negative results.

Despite the discouraging comments thus far, the loss of water which occurs when cherts are heated is a very significant factor in the alteration of the rock. With the removal of the intercrystalline water, the microcrystals become firmly cemented. Thus when a fracture occurs it passes through rather than around the individual crystals. In other words, the stone breaks more like glass than a rock aggregate even though the same microcrystalline structure and texture are still present. This, in turn, explains the increase in vitreousness of the fractured surface. Chert is composed of microcrystals which constitute the mineral phase known as chalcedony. Mineralogically, chalcedony is waxy or greasy in luster but individual faces of this mineral are normally not seen because the crystals are anhedral (no definite shape or orientation), usually subequidimensional, and microscopic. Therefore, when fracture occurs, especially if the rock is coarse grained, the

fractured surface is dull due to refraction and poor reflection. After heating, the fractured surface is vitreous due to the greater transmittance of light which occurs when the fracture passes through successive microcrystals and intercrystal spaces revealing the intragranular nature of quartz.

If the material is fine grained, it seems to alter slightly faster which may be because, even though it contains the same overall amount of water, there is less moisture being removed from any given interstitial area. There seems to be no correlation between crystal size and the temperature at which alteration occurs in Florida cherts. There does seem to be some correlation between the composition of the chert and the length of time as well as the temperature at which alteration occurs. The fact that Florida cherts are formed as limestone replacements was discussed in the section on Materials. Some of the materials tested appeared to react differently than others in that they did not respond at the same temperatures or time periods; also they did not decrepitate or explode. Except for the area directly under the cortex which always seems to be very siliceous, a satisfactory change did not occur in some rocks at the testing temperatures which usually effected a change.

When temperatures are raised slowly and left at either 350°C or 400°C a change occurs which is very apparent when the sample is subsequently flaked revealing a vitreous fractured surface. In addition, it is definitely easier to manufacture stone implements (see Figure 6a). This statement

is not based entirely on intuition (see section on Strength Tests). It remains to be explained, however, why it is that if the temperature is raised too rapidly, explosion occurs. The original weight loss at 100°C is due to the removal of adsorbed water held on the surfaces of the individual microcrystals. After this initial large water loss, the weight remains quite stable until 350°C. Prior to this, the temperature probably has not been high enough to remove the chemically bound water held in the intercrystalline areas. I suspect that the following happens: 350°C is probably a sufficiently high temperature for alteration to take place if this temperature is sustained for a long period of time (see Table 6). If the temperature is raised to 400°C prior to the gradual removal of the chemically bound water, explosion results because the alteration proceeds too rapidly. Vitrification is always evident, at least to some extent, on the fractured surfaces which result from these explosions. Therefore, some change must occur almost immediately when a critical temperature is reached even though the material blows up at the same time. There does not seem to be any previous study dealing with this problem adequately. Preston (1926) in his article on the rupture of glass discusses the fact that glass will fracture when alternately subjected to heat and cold. But his work mostly describes the type of fracture which will occur depending on the stress to which the material is subjected. Perhaps the answer lies in the following statement:

> . . . principles of surface chemistry . . . . When a crystal consists of highly polarizable anions of large size, together with small, highly charged cations, then the anions will be pushed to the surface of the crystal and the cations will be recessed. Thus in a microcrystal of quartz oxygen ions predominate at the surface, while the silicon ions are depressed. It is believed that each microcrystal of quartz then has a negatively charged "skin," and effectively repels adjacent randomly oriented microcrystals . . . fracture probably takes place between the polyhedral blocks because of the surface repulsion forces (Folk and Weaver 1952: 507-8).

When the rocks are subjected to critical temperatures, there may be a change in the position of oxygen and silicon ions as described above. If the ions become too excited when temperature is applied rapidly, explosion may take place. This may also explain the clicking sounds which occur and often result in exfoliation when the material is rapidly cooled. Perhaps the reason these clicking sounds aren't heard when the material is removed after a sustained period is because the process is completed and no further change is taking place.

> Failure was accompanied by peculiar "clicks." These "clicks" were heard early in the cooling period and never during the heating period, when the rate of temperature change was lower. When the cups were heated to a slightly higher temperature, no higher percentage of failures was observed (Pressler and Shearer 1926: 307).

Material that has been heated to 500°C is no easier to flake than that left at 350°C for prolonged periods (see Fig. 6b). A number of samples were heated to 600°C; subsequent chipping of these specimens did not reveal any further change either. However, point tensile strength tests, to be discussed later, do not support this observation since there is increased strength loss with increased temperatures under point tensile

load. In addition, while there is no apparent increase in flaking ease, quite often attempts to chip flint materials that had been heated to 500°C-600°C or had been heated to 350°C-400°C and removed immediately from the hot oven, resulted in a lateral snap due to end shock. This fracture did not occur at the point of impact. The fractured surface often resembles that illustrated in Figure 8. Flint-knappers are familiar with this type of failure since it occurs when a substantial blow is imparted to a rock whose mass is not adequately supported to absorb the shock. With these heated materials, however, failure occurred when only slight pressure or percussion was applied.

The composition of the chemically bound water may not be identical to water as we normally think of it, that is H_2O. If the hydrogen and oxygen ions are dissociated and the oxygen plus part of the hydrogen are given off when a certain temperature is reached, the remaining hydrogen ion with its positive charge may hook up with the negatively charged oxygen "skin" of the microcrystal and serve as the binder. Or if the depressed silicon ion is agitated, it may hook up with an oxygen ion from an adjoining microcrystal and thus form the bond in that way.

Pressler and Shearer (1926) offer some suggestions concerning the reactions of various types of flints when heated. Their article is concerned primarily with flints used in the ceramic industry but may hold some applicable clues. Since temperatures of 1400°C-1700°C (>3000°F) are

needed to transform microcrystalline structures to a non-crystalline form, the vitrification which occurs when flint materials are heated to only 350°C must be accounted for in another way.

> The impurities in flints generally act as fluxes, due to eutectic developments. . . .
>
> . . . Fe_2O_3 was found in all flints varying from 0.03 to 0.12 per cent. While iron is objectionable as an impurity because of its discoloring effects, the small percentage present in the flints would probably be of no significance in a body composition. Some of the American flints are free from CaO, and others contain generally less than the French flints. The loss on ignition varies from 0.13 to 0.85 per cent and represents CO_2 dissociated from $CaCO_3$, and adsorbed or chemically combined water (Pressler and Shearer 1926: 292-3).

If the impurities (or combinations of impurities) contained in Florida cherts are serving as fluxes (substances promoting fusion) to fuse a thin surface film of the cryptocrystals, then change will occur when the melting point temperatures for these impurities are reached (eutectic development). If this is the case, answers are found for two puzzling problems: (1) why vitrification occurs at such a low temperature (350°C), and (2) why some materials do not respond at the same temperatures--the melting points of the fluxes are probably different. If the percentage of calcium is quite high, this also explains why certain materials do not make a desirable change despite the temperature or length of the heating period. Calcium will serve as a flux if present in small quantities, but prevents the desired reaction from occurring if present in large quantities. Three Florida cherts which responded

differently when heated were submitted to the Soils Department at the University of Florida for Atomic Absorption Spectrophotometer analysis. The results of this analysis are given in Table 7. It is not known what percentage of calcium is necessary to prevent the desired reaction but it is interesting to note that Ocala chert and High Springs chert had considerably higher percentages of calcium than the two Johnson Lake specimens. The Ocala and High Springs cherts consistently reacted differently than Johnson Lake and other Florida materials tested in that the expected change did not occur within the same temperature and time ranges.

Figures 7, 8, and 9 illustrate various types of fractures. Figure 7 shows an example of a potlid fracture which occurred only when the materials were heated too rapidly. Figure 8 shows the type of fractured surface which often occurred when the samples were removed from a hot oven to a cool environment. The specimens in Figures 7 and 8 have broken with a conchoidal fracture typical of flint materials. Figure 9 is a thinning flake, intentionally struck from an obsidian core. In addition to the conchoidal fracture, this last specimen possesses a well-defined bulb of percussion not present in Figures 7 and 8. Figure 9 serves as a reminder that a microcystalline rock will break with a conchoidal fracture but no bulb of percussion will be evident unless impact has taken place.

The results of the heating experiments establish quite clearly that crystal boundaries may be a disturbing

TABLE 7

RESULTS OF ATOMIC ABSORPTION SPECTROPHOTOMETER ANALYSIS

Sample	Percentage					
	Mg	Fe	Ca	P	Mn	K
Johnson Lake (fine grained)	.010	.250	.033	.003	.015	.040
Johnson Lake (coarse grained)	.008	.400	.045	.016	.010	.020
High Springs	.011	.1115	.095	.033	.010	.042
Ocala	.040	.290	6.600	.040	.025	.050

Figure 7.--Example of a Potlid Fracture which Often Occurred when Florida Cherts Were Subjected too Rapidly to 400°C Temperatures.

Figure 8.--Examples of a "Crenated" Fracture which Often Occurred when Specimens Were Removed Directly from a Hot Oven to a Cool Environment.

Figure 9.--Specimen Depicting Intentional Fracture with Bulb of Percussion and Typical Fractured Surface Emphasizing that Impact Has Occurred.

influence when attempting to predict fracture. Therefore, the more glass-like the material, the more predictable the fracture. Heated cherts are more glass-like and fractures are not only more predictable but easier to execute.

Iron Content of Florida Chert

The incorporation into chert formations of concentrations of iron which impart a pink to red color upon heating is usually because of secondary enrichment resulting from the mobility of iron in iron-rich paleosoils or bog deposits. In some of the Lower Miocene deposits, however, another situation exists in which iron seems to have been incorporated at the time of silicification which resulted in the chert formation. In addition, although they are rare, bright pink colors (ca 5 R 7/4) (Munsell 1946) are sometimes found in cherts imbedded in terra rossa residual soils on Ocala Limestone. These cherts are dull or earthy in appearance and do not resemble cherts which have been thermally altered (H. K. Brooks, personal communication).

Color change takes place between 240°C and 260°C in Florida cherts. Figure 10 illustrates the variation which results depending upon the amount of iron present in the sample. Unfortunately, true color representation was not attained but an analysis of the iron content (see samples in Figure 10b) revealed that samples changing from 10 YR 6.5/2 (between very pale orange and pale yellowish brown but nearer the latter) to 10 R 4/4 (between pale reddish brown and dark reddish brown) contained 4000 ppm

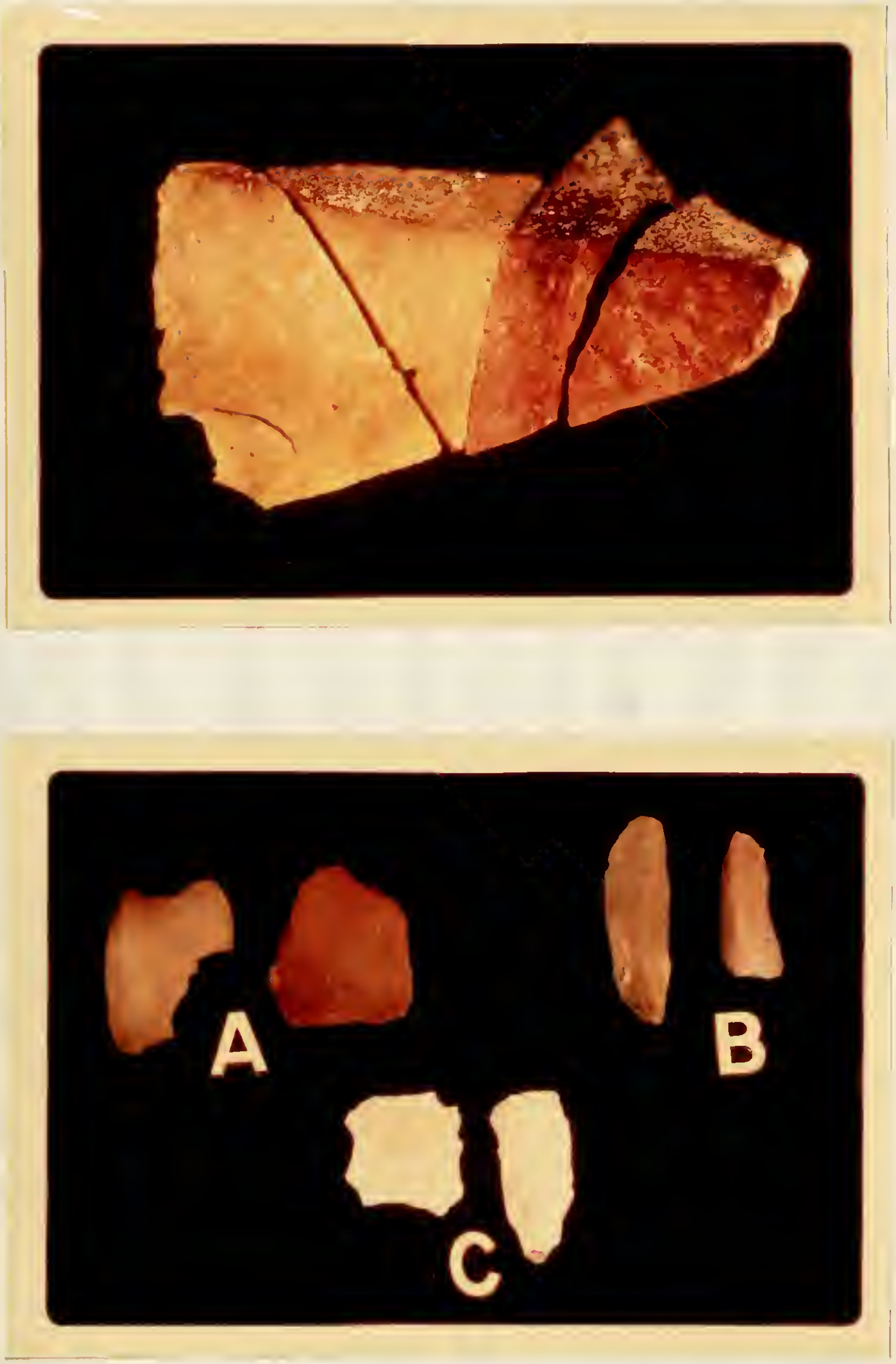


Figure 10.--Heated Specimens and Unheated Controls Illustrating Degree of Color Change Depending upon the Amount of Iron Present in the Chert.

(.40%) of iron. Those changing from N 6.5 (between light gray and medium light gray) to 5 R 7/2 (between grayish pink and pale red) contained 2500 ppm (.25%). Those exhibiting no color change contained 1100 ppm (.11%). It should be emphasized that:

1. Color change occurs only because of the oxidation of iron and will not occur if no iron is present. Many samples of Florida materials did not change color.

2. The temperature at which color change occurs (240°C-260°C) is not synchronous with the change to vitreous luster in Florida materials--ca 350°C-400°C for sustained periods.

3. Therefore, color change cannot be used as a reliable criterion (at least for Florida cherts) in ascertaining if materials recovered from archaeological sites have been intentionally thermally altered. But if a combination of vitreousness and color change occur frequently on artifacts or waste flakes as is the case in Florida, the assumption that man was subjecting the chert to temperatures sufficiently high to cause a change prior to final chipping is valid.

Strength Tests

Preparation of the Samples

Uniform-sized samples were prepared in order to determine, by standard rock mechanics tests, whether any differences in compressive and point tensile strength exist between heated and unheated specimens.

Initially, twelve 1-inch cubes were prepared, each weighing approximately 42 grams. Using a diamond blade, it was necessary to saw the stone as accurately as possible to the desired dimensions and then laboriously grind the samples to exactly one inch. Any deviation would have rendered the results unreliable. Since this method was extremely

time consuming, a coring device was used which greatly reduced the time needed to prepare samples. Using a drill press as a basic piece of machinery (Figure 11a), a Corematic diamond core bit having an inside diameter of 1 inch ± .005 with a 6-inch core barrel and 3/8-inch diamond penetration was constructed by Anton Smit and Co., Inc., New York. This is a variation of a Corematic drill used to cut holes in glass. The chert nodule to be cored was clamped into position as illustrated in Figure 11b.

The diameter of these cored samples was exactly one inch. The cored samples that were to be used for compressive tests were then sawed as close to one inch in length as possible and ground to precisely one inch. This reduced the number of surfaces to be ground from six for the cubed samples to two for the cored samples. The cored samples that were to be used for point tensile strength needed no additional preparation.

Compressive Tests

Of the original twelve 1-inch cubes, six were heated to 500°C as described in the section on Heating Experiments and six were kept as unheated controls. One sample each of heated and unheated obsidian, silicified coral, and chert from a quarry 3 miles north of Ocala[1] were used for compressive tests. Compressive strength data obtained from the cubed samples of Ocala chert were unreliable due to error in testing; these were discarded. Subsequently, two cored

[1]To identify this material as to location it will hereafter be called Ocala chert.

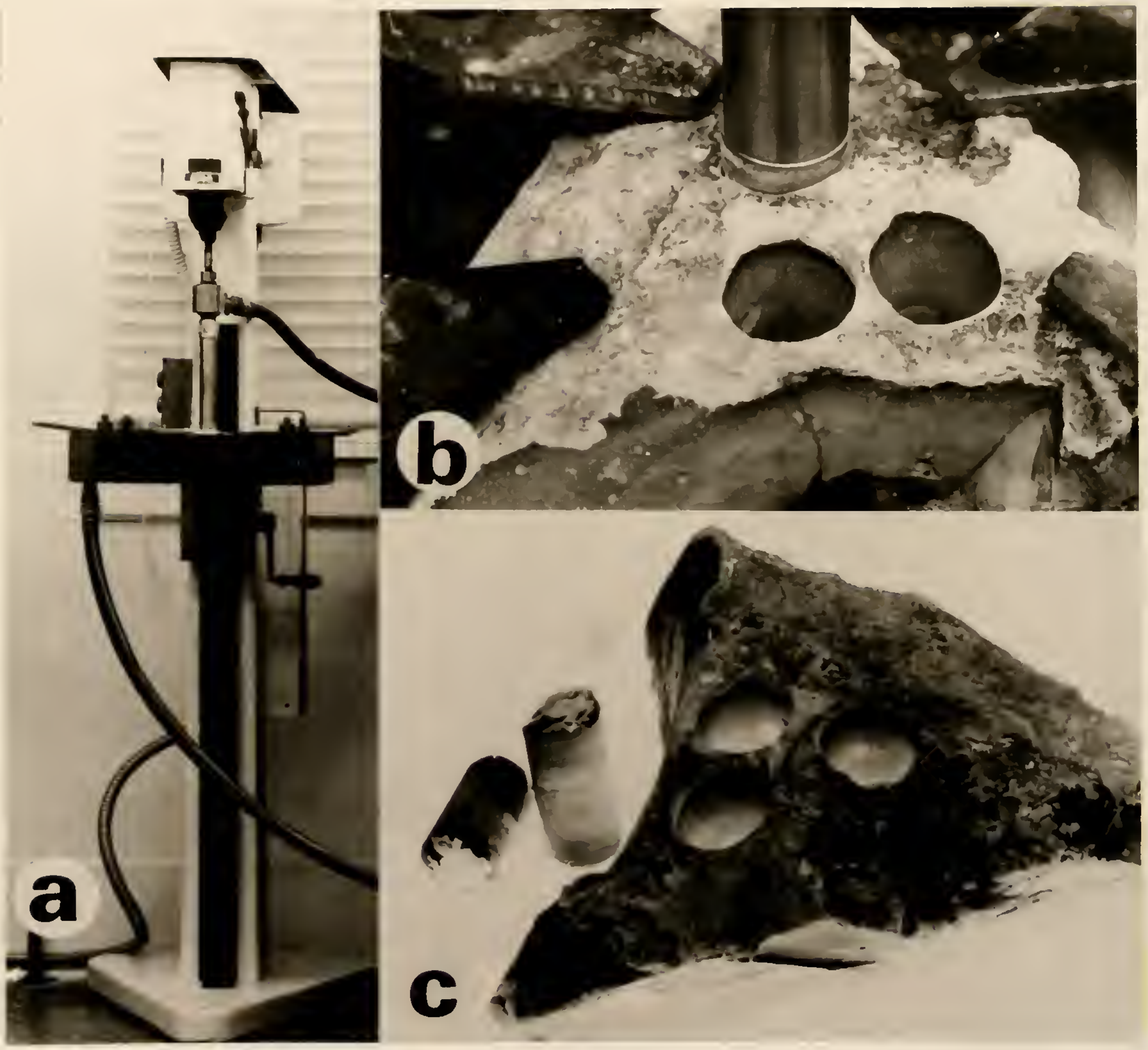


Figure 11.--Composite Illustration of: (a) Drill Press, (b) Corematic Diamond Core Bit, and (c) a Chert Nodule with Cores Removed to be Used for Strength Tests.

samples of Ocala chert, 1 inch in length, were prepared. One of these was heated to 400°C and the other was kept as an unheated control. Both cubed and cored heated samples had been removed from the oven immediately at the end of the heating period without first being allowed to cool. The significance of this procedure will become apparent later.

Arrangements were made with the Civil Engineering Department at the University of Florida to test the strength of these materials. The equipment used was a 300,000-lb capacity Riehle Universal testing machine with hydraulic type loading and five ranges. The results are given in Table 8. The cross sectional area of the cored samples was standardized to 1 inch as follows:

$$A = \pi r^2$$

$$A = .785$$

The figures resulting from the compressive strength tests for the cored samples were then divided by .785 thus expressing the force in pounds of pressure per square inch.

In another test, cored samples of obsidian and High Springs chert were prepared for compressive strength tests as described above. One sample of each of these materials was heated to 400°C and allowed to cool in the oven. One sample of each was retained as an unheated control. The results are given in Table 9. See Figure 12 which graphically illustrates the data presented in Tables 8 and 9.

The compressive tests yielded the following results. From the data given in Table 8, it is apparent that when

TABLE 8

RESULTS OF COMPRESSIVE STRENGTH TESTS WHEN HEATED SPECIMENS ARE SUBJECTED TO A COOL ENVIRONMENT WHILE STILL HOT

Sample	Unheated Control (psi)	Heated (psi)	Strength Loss (%)
Obsidian X	50,100	30,000	40
Silicified coral	43,900	25,000	43
Ocala chert	52,994	31,720	40

TABLE 9

RESULTS OF COMPRESSIVE STRENGTH TESTS WHEN HEATED SPECIMENS ARE ALLOWED TO COOL IN THE OVEN

Sample	Unheated Control (psi)	Heated (psi)	Increase in Strength (%)
Obsidian Y	31,100	39,600	25
High Springs chert	25,100	42,200	40

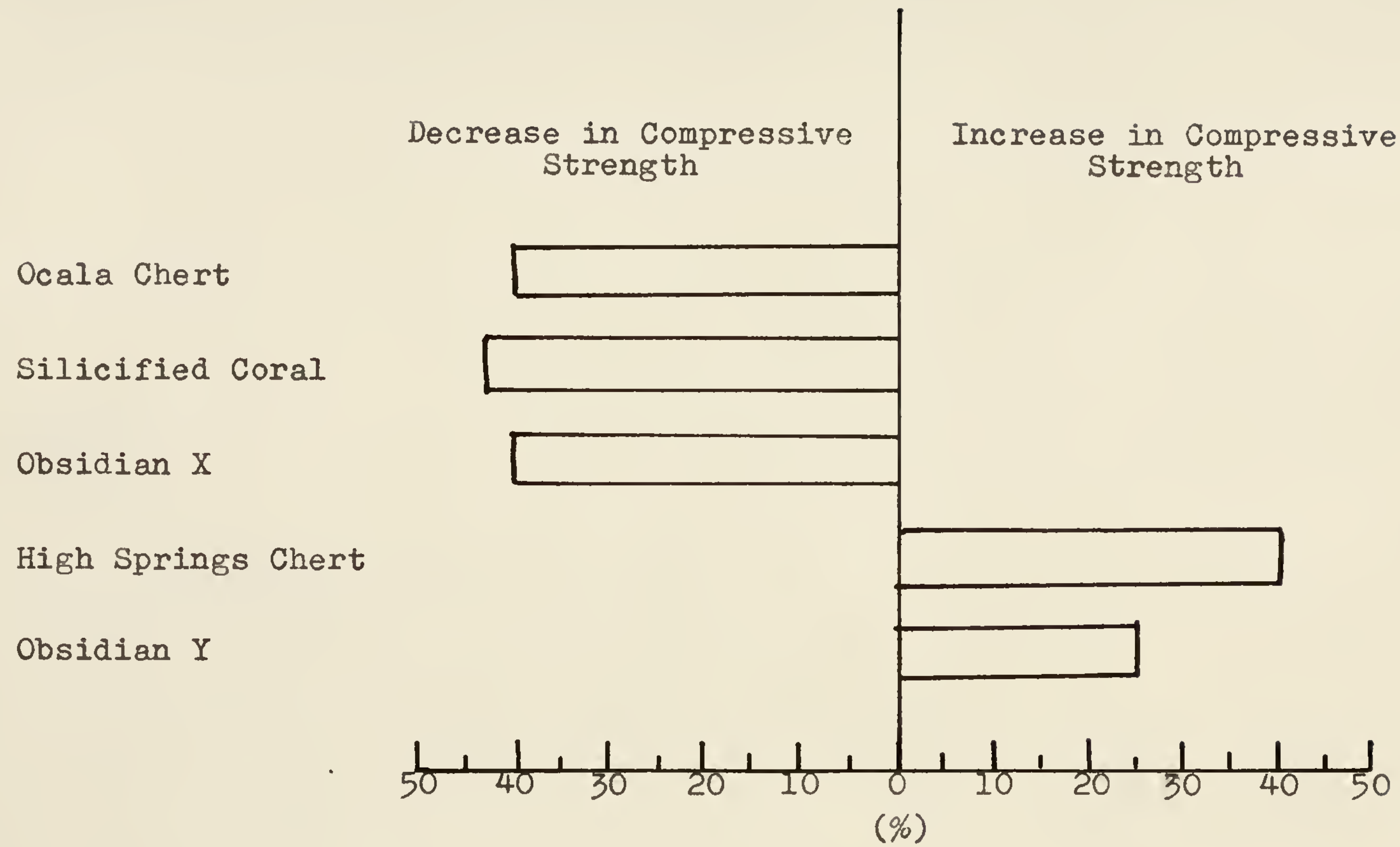


Figure 12.--Decrease or Increase in Compressive Strength over Unheated Controls of Heated Specimens; Samples Showing a Decrease Had Been Removed Immediately from a Hot Oven while Samples Showing an Increase Had Been Cooled before Removal.

siliceous materials are heated to temperatures of 400°C-500°C for sustained periods and removed while hot, certain stresses occur which cause a reduction of strength when compared to the unheated controls. The three unheated varieties withstood forces ranging from 44,000 to 53,000 psi (rounded figures) while the heated samples of the same materials ranged from 25,000 to 32,000 psi. This represents approximately a 40% reduction of strength in the heated samples. On the other hand, when the heated samples were allowed to cool in the oven, as shown in Table 9, there was an increase in strength amounting to approximately 25% for obsidian and 40% for the High Springs chert. Cohering the material without the introduction of stresses allowed the heated samples to resist failure for a longer time than unheated counterparts or heated specimens that had been stressed by sudden subjection to a cool environment.

No comparison should be made between the pounds of pressure per square inch recorded in Tables 8 and 9. The experiments were conducted several months apart, the equipment was operated by different individuals, and the rate of load was not observed for the samples removed from the hot oven and their controls. The rate of load for the Table 9 samples was approximately 20,000 pounds per minute. In addition, and most importantly, the samples were different. The obsidian samples were from different locations and therefore probably of different ages and depositional situations. The reader is reminded that man has classified

both of these specimens as obsidian based on certain arbitrary attributes shared by both; the use of other attributes may have resulted in a different category for each. Of significance here is the reversal of the amount of pressure needed for fracturing the material depending on whether the rock was removed from a hot oven or was first allowed to gradually cool in the oven.

Point Tensile Tests

A jig was machined to apply stress to the cores as described by Reichmuth (1963). This jig was used in connection with the Riehle Universal testing machine described previously. The jig was constructed to apply point tensile load to the curved surface of the cylindrically cored specimen with the long axis of the specimen placed horizontally and at right angles to the loading jig. The jig was constructed so that the point compressive loads were applied through small diameter steel hardened dowel pins with rollers manufactured by Holo-Krome of West Hartford, Connecticut (see Figure 13). The cones of percussion induced at the points of application of compression produce internal tensile stresses perpendicular to the load axis.

Cored samples, one inch in diameter of varying lengths were prepared as described earlier. In the first experiment, two cored specimens of Ocala chert were heated to 400°C for 24 hours and removed immediately from the hot oven at the end of the heating period; two samples were retained as unheated controls. The results of this

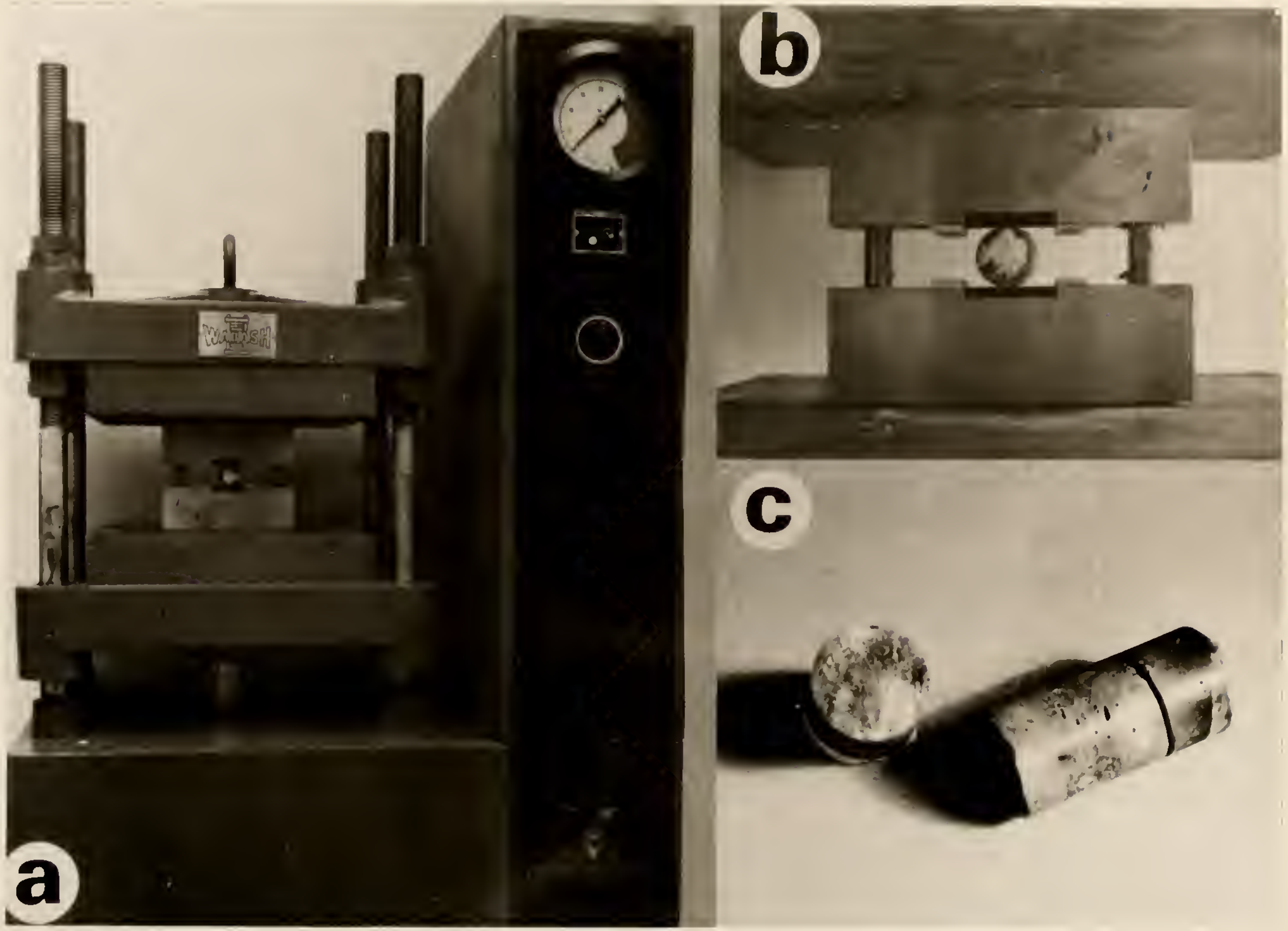


Figure 13.--Composite Illustration of: (a) Compressive Strength Testing Machine, (b) Close Up of Jig with Core in Position, and (c) Core Split by Application of Point Tensile Load.

experiment were as follows. The unheated Ocala chert withstood forces averaging 2700 psi while the heated Ocala chert withstood forces averaging 1500 psi. This represents a reduction in force by 45% needed to break the material. The point load tensile strength was computed from the following empirical expression given by Reichmuth (1963):

$$T = .96\frac{F}{D^2} \text{ where}$$

T = tensile strength

F = total failure load in pounds

D = core diameter in inches

The information to be gained from point tensile tests was considered pertinent to this investigation because the amount of force used to induce failure of the material by point tensile stress is essentially the same as the strength needed to induce fracture when manufacturing lithic tools from siliceous materials by either percussion or pressure methods. Therefore, extensive experiments were set up to test the point tensile strength of differing materials in various ways.

1. Four samples of obsidian, High Springs chert, and Johnson Lake chert were tested as follows and allowed to cool in the oven:

1 sample of each used as controls
1 sample of each heated to 300°C for 24 hours
1 sample of each heated to 350°C for 24 hours
1 sample of each heated to 400°C for 24 hours

2. Two samples of obsidian and of Johnson Lake chert (from same core as Johnson Lake chert used above)

were heated to 300°C and 350°C left for 24 hours, and then removed immediately from the oven.

The results of these tests are given in Table 10. In all samples except obsidian for which the data were not consistent, there is an increasing reduction in strength with increase in temperature. In addition, though admittedly the data are scant, there is an even greater increase in reduction in strength when heated samples are removed immediately from the hot oven.

If a comparison is made between the results obtained for compressive tests with those for point tensile, a discrepancy seems to exist. Under compressive strength when samples are allowed to cool in the oven they resist failure longer than unheated controls. The results of point tensile tests, however, revealed a significant reduction in the time and load necessary for failure to occur in heated samples regardless of whether they were removed from the oven while hot or allowed to cool in the oven. This seeming paradox is easily explained. The binding of the microcrystals which occurs when the rock is heated adds compressive strength through cohesion to the structure. The increase in homogeneity which increases strength under compression is the very factor which descreases point tensile strength: (1) the individual microcrystals are bound more firmly together; (2) therefore, when the flaw is introduced which is preliminary to and necessary for fracture to occur; (3) failure takes place more readily because the specimen fractures more like glass than a rock aggregate. The added decrease in

TABLE 10

RESULTS OF POINT TENSILE STRENGTH TESTS[a]

Sample	Unheated Control	300°C	350°C	400°C	Total Strength Loss (%)
		Allowed to Cool in Oven			
Obsidian	1872 1944	2640	1536 1416	2232 1368	--[b]
High Springs chert	2568	2448	2136 1680	1584	39
Johnson Lake chert	3072	2472 1512	1680 2016	1392 1392	55
		Removed from Hot Oven			
Obsidian		1123	1104 2670[c]		
Johnson Lake chert	3072	1344	1152		63

[a]Rate of load was approximately 2000 lb/min.

[b]The results are not consistent.

[c]It is difficult to explain this anomaly with what otherwise appears to be a significant difference.

strength which occurs when specimens are removed immediately from the hot oven is due to stresses resulting when the material is exposed too rapidly to a cool environment.

X-Ray Diffraction Pattern

Twenty-six x-ray diffraction patterns were run on 13 different samples of heated and unheated cherts. No consistent differences could be detected indicating that no change in the crystal lattice occurs (see Figure 14). The slight difference shown is well within the range of experimental error. If the crystals were coarser, the peak would be higher; if the crystals were finer, the peak would be lower.

Differential Thermal Analysis (DTA)

The method of studying materials by differential thermal analysis consists of heating a small, finely ground sample at a constant and rapid rate, and recording by suitable means the endothermic and exothermic effects. A differential thermocouple is used to detect these effects. One of the thermocouple junctions is placed in the sample being studied and the other is set in a thermally inert substance that is undergoing the same heat treatment as the sample (Berkelhamer 1944). The electric current generated by the differential thermocouple is amplified and recorded. Endothermic peaks result if the sample is taking on energy.

Samples were prepared of pure quartz, pure opal, heated and unheated Johnson Lake chert, High Springs chert,

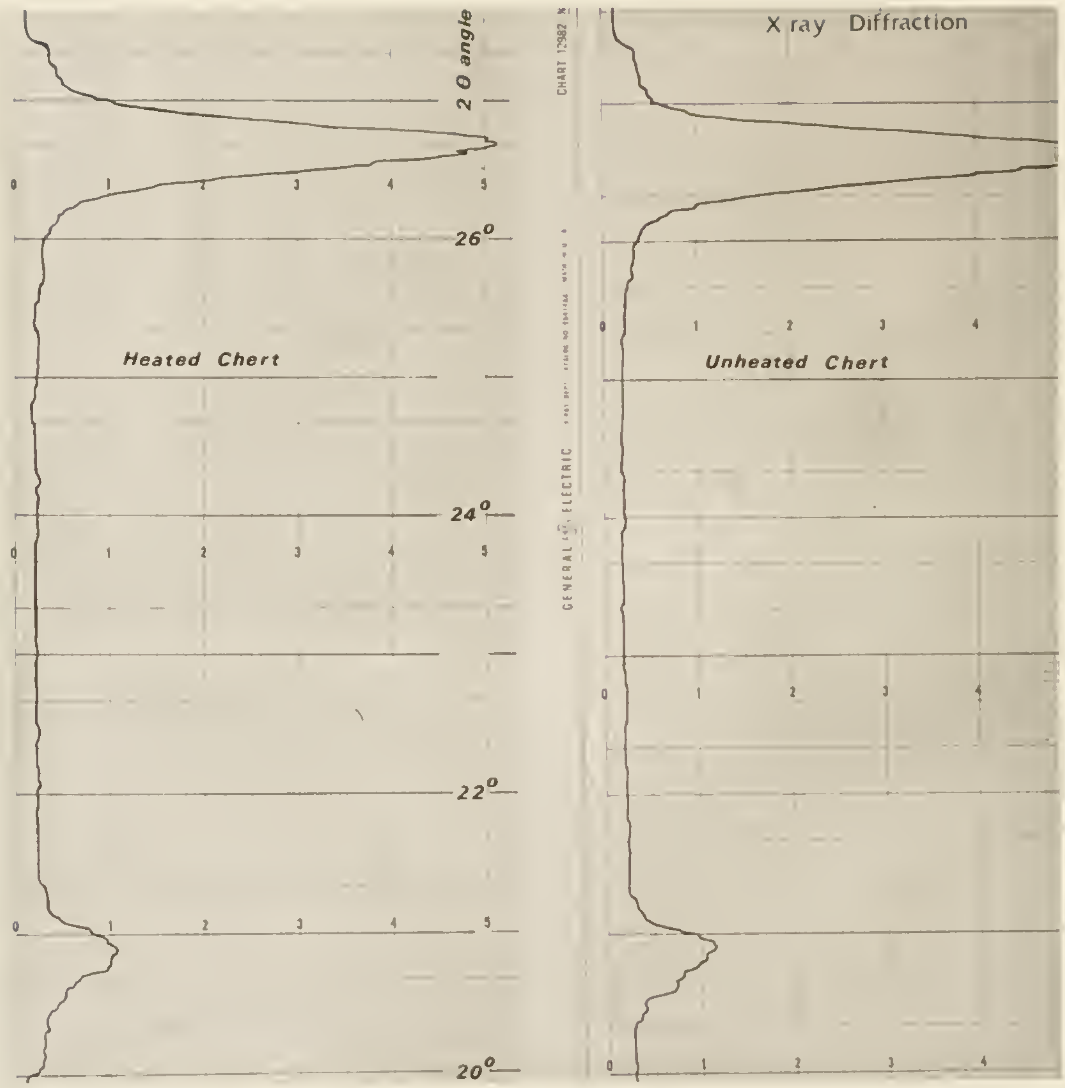


Figure 14.--X-Ray Diffraction Pattern Illustrating that No Change Occurs in the Crystal Lattice when Florida Cherts Are Subjected to Critical Temperatures.

Ocala chert, English flint, and obsidian. Arrangements were made with the Soils Department at the University of Florida to use the differential thermal analysis equipment. The results of this experiment were encouraging since they substantiated data obtained from the weight loss experiment. As the sample takes on energy (heat), water is driven off. This was apparent by the endothermic peaks at 112°C as shown in Figure 15 for opal, Johnson Lake chert not previously heated, Ocala chert, and English flint. This was due to the removal of the adsorbed water held on the surfaces of the microcrystals. In the pure quartz sample, the already heated chert sample, the High Springs chert sample, and obsidian there is no peak at 112°C. These materials have already been devolatilized or there is no moisture to drive off. The only anomaly that seems to exist is that the chert from High Springs which is Oligocene in age did not show a peak at 112°C. This material is more coarsely crystalline than much of other Florida material. This fact is borne out by the quartz peak at 573°C occurring with this sample and the pure quartz sample. This peak represents the alpha-beta change in the crystal lattice, and it is immediate as the sharp peak indicates. This inversion reaction is reversible in heating and cooling.

To check the validity of the endothermic trend beginning about 350°C-400°C (see Figure 15), additional samples were prepared and tested as before. These materials were rerun to determine if the endothermic trend indicating an

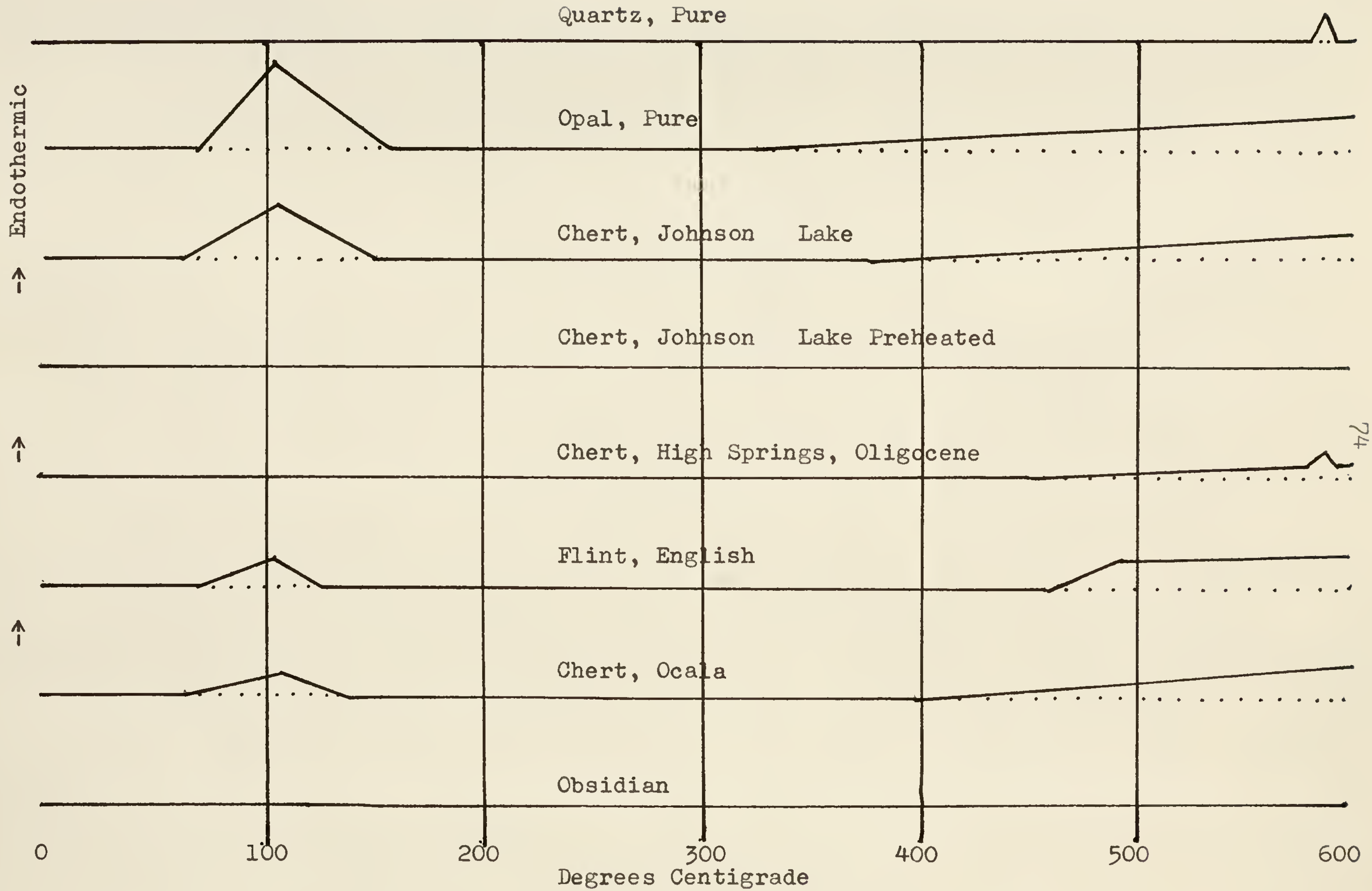


Figure 15.--Results of Differential Thermal Analysis

increase in energy being absorbed may have been due to an instrumental error. Except for the absence of the peak at 112°C, the rerun materials exhibited the same type of curves as were initially observed. The sensitivity of the recording galvanometer of the Soils Department had been adjusted for the detection of the characteristic peaks of minerals whose thermal reactions are of greater magnitude. Thus, within the framework of tests available to this experiment, the curves seem valid.

Petrographic Analysis

A total of 45 petrographic sections were ground to approximately 10μ to show the internal structures of chert, especially grain size and orientation, intergranular relationships, and cracks. A careful search of the slides at a magnification of 100X to detect evidence of differences between heated and unheated specimens failed to reveal anything significant. Some of the slides suggested that heated materials had more fractures which were more open and more oriented. Due to the heterogeneity of chert, however, it is hazardous to draw any conclusions from the petrographic study other than the fact that there is no change in size of the individual crystals or their orientation when cherts are heated to temperatures of 350°C-400°C. This statement is in agreement with the findings of Tullis (1970: 1344) who reports that Dover flint even when subjected to axial compression revealed no preferred orientation when loaded at 400°C under a differential stress of 3 kb for 20 minutes.

Three additional slides of Johnson Lake chert were ground to 3µ, which is standard petrographic thickness, to determine if thermal stresses could be detected which may have been generated within the grain by the application of heat. At a magnification of 430X no differences were observed for an unheated control, a specimen heated to 400°C and allowed to cool in the oven, and a specimen heated to 400°C and removed immediately from the oven to a cool environment (see Figure 16). Thus, despite the fact that strength tests revealed a significant reduction or increase in strength between heated and unheated cherts, and that point tensile tests indicated there is an even greater reduction if materials are removed immediately from a hot oven, these changes are not observable petrographically.

Determination of Specific Surface Area

The method involved in determining the surface area consisted of the gas absorption technique in which helium is used. The data were analyzed by an Orr Surface-Area Pore-Volume Analyzer. Four samples were submitted: High Springs Oligocene chert which is coarse grained and gave a quartz inversion peak on differential thermal analysis, Johnson Lake Eocene chert which is fine grained, and Eocene chert from north of Ocala--both heated and unheated. The results of the tests on the unheated cherts were consistent with their grain size and packing. The least specific surface area was 4.64 M^2/g for the High Springs chert; 5.39 M^2/g for the Johnson Lake chert; and 4.86 M^2/g for the Eocene chert

unheated

heated

Figure 16.--Petrographic Sections Showing No Detectable Change in Heated vs Unheated Florida Chert (Scale: 1 inch = 1,500,000 Å).

from Ocala whose grain size is intermediate of the three samples. Most significantly, the Eocene chert from Ocala that had been heated showed a marked reduction in the granular surface area: 1.90 M^2/g compared to 4.86 M^2/g for its unheated counterpart. This represents approximately a 60% reduction in the granular surface area of the heated chert. There is no question but that the microcrystalline surface area has been reduced in the heated specimen. This was due to the reduction of the intergranular pore radii. In other words, porosity had been decreased due to a fusion or intergrowth of the grains.

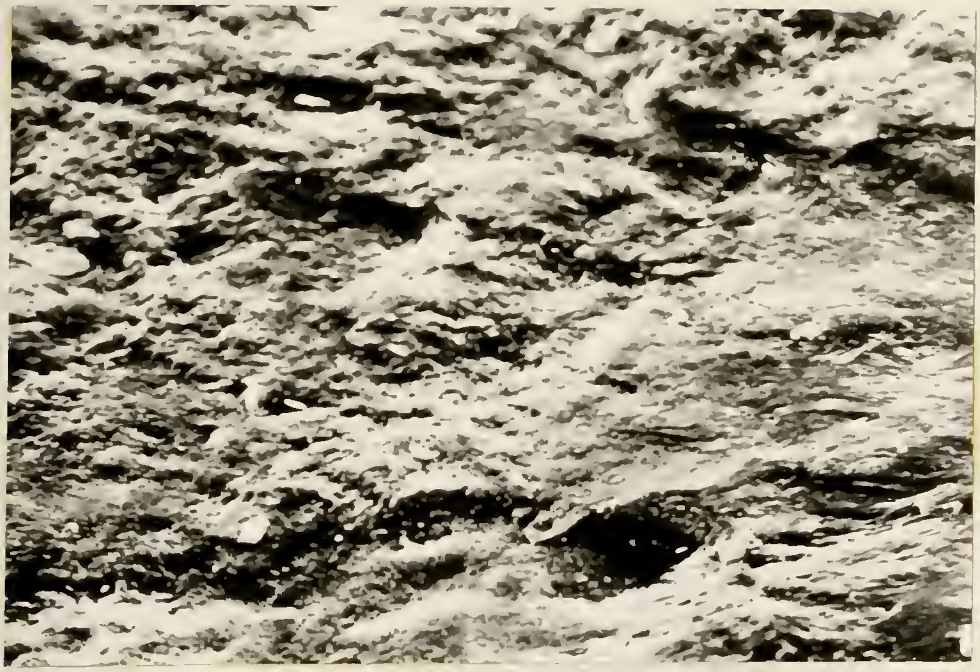
A computer printout showed the size of the pore space distribution and surface area distribution relative to percentage of occurrence. The plot for Ocala unheated chert showed the minute size of the original pores: 27.81% of the pores were 23μ or smaller in size whereas about .14% were 370.8μ.

Scanning Electron Microscope

The scanning electron microscope (SEM) is used to study surface morphology (Krinsley and Margolis 1968). Small flakes not exceeding 1/8 x 1/2 inch were pressed from unheated and heated cherts. The samples were cleaned with acetone and mounted with Scotch tape on an SEM specimen plug. Three or four specimens were mounted on each plug. The freshly fractured surfaces were exposed for scanning. The specimens were then coated with gold "in order to make the surfaces a better conductor for the

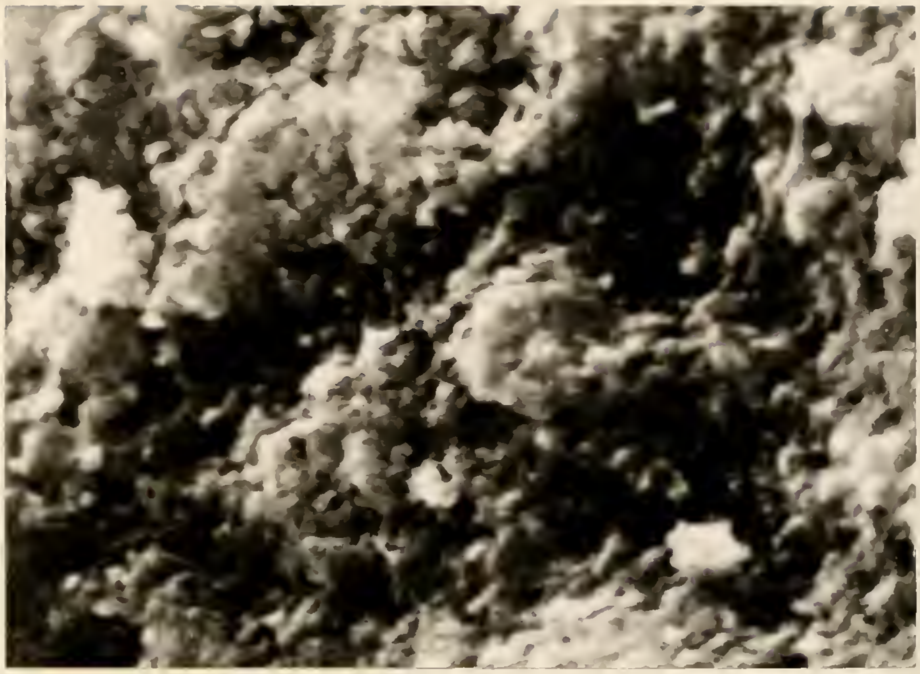
electron beam; the technique does not eliminate or create any additional surface features" (Krinsley and Margolis 1968: 458). Heated and unheated samples included silicified coral, Ocala chert, High Springs chert, and fine and coarse grained Johnson Lake chert. The specimens were placed in a Cambridge Stereoscan electron microscope housed in the Department of Metallurgy on the University of Florida Campus. The entire fractured surface of each of these specimens was scanned. Figures 17 and 18 show the typical surface topography, dramatically illustrating the changes which occur when cherts are heated. Figure 17 is of Johnson Lake chert (1000X); Figure 18 is of silicified coral (12,200X). In the unheated specimens, the individual grains are seen looking like so many bread crumbs. Some fracturing has occurred through the individual grains but more frequently the fracture goes around the grains. In the heated specimens, the fractures pass through most of the individual grains; that is, the individual grains are actually split, and the fractures continue on passing through the interstitial areas which are now more firmly cemented. In other words, when fracture occurs it alternately splits and passes through succeeding crystals and intercrystal areas in its path until it terminates. This accounts for the smooth surface in the heated specimens.

unheated



heated

Figure 17.--Surface Topography of Unheated and Heated Johnson Lake Chert Samples as Viewed by the Scanning Electron Microscope (Scale: 1 inch = 254,000 Å).



unheated

heated

Figure 18.--Surface Topography of Unheated and Heated Silicified Coral from Florida as Viewed by the Scanning Electron Microscope (Scale: 1 inch = 20,320 Å).

ARCHAEOLOGICAL APPLICATION

If an archaeologist suspects that the chipped stone remains he recovers from a site have been thermally altered, it would be desirable to subject these materials to a standardized test in order to eliminate the "guess" factor. This is important since thermal alteration presents information concerning not only stone technology, but contributes ultimately to a greater understanding of prehistoric man's behavioral patterns. This becomes even more valuable if an investigator is working on time levels where only stone artifacts have been preserved.

The following discussion involves a review of the many experiments conducted throughout the course of this study to determine if one or more of these might feasibly be applied to site materials. For a fuller account of these experiments, the reader is advised to refer to the section on Methodology.

Weight loss cannot be used as a criterion to determine if specimens had been thermally altered because experiments demonstrated that heated specimens tended to take on moisture again. In other words, if an archaeologist weighed, heated, and reweighed outcrop materials along with site materials to check differences in weight loss he probably would find no significant difference. And even if

he did, he would have to use the results very cautiously since he could not be absolutely sure that his site material was identical with the outcrop material. Chert will vary considerably even within the same nodule. Differential thermal analysis probably would also reflect this tendency of the material to take on moisture.

If stone remains are found with "potlid" fractures or fractures minus bulbs of percussion, it may be said that the stone had been fractured due to expansion or contraction resulting from heat, but there would be no sure way of determining if the exposure to the heat had been intentional or because of a forest fire or hearth situation.

Standard rock mechanics tests might be of value to test site specimens with outcrop materials, but it probably would be impossible to recover large enough specimens from sites to prepare samples whose dimensions must be precisely accurate in order to assure the validity of the results. Thus, this method succeeds in theory but fails in practical application.

Petrographic analysis revealed no change in the size, shape, or orientation of the individual microcrystals. This was borne out by the x-ray diffraction pattern which showed no change in the crystal lattice between heated and unheated materials.

The vitreousness of a surface that has been flaked subsequent to thermal alteration offers, perhaps, the most valid indication that this method has been employed.

Vitreousness may not be completely reliable, however. Several broken specimens were selected from preceramic levels (>2000 BC) of a site (A-356) in Florida. These specimens were chosen because their outer surfaces were extremely lustrous and some exhibited a pinkish cast. It was suspected that all had been thermally altered. Small flakes were pressed from each specimen. One specimen which had an especially greasy luster on its outer surface was very difficult to flake and the freshly flaked surface was dull. This was a disappointing development, because it was hoped that by removing chips from field specimens, it might be determined that vitrification which penetrated the entire mass of test materials is a permanent change and might be useful for archaeological interpretation especially if local outcrop materials are not naturally vitreous. This specimen had either patinated subsequent to thermal alteration resulting in a replacement of the internal luster, or soil conditions due to long burial had operated to form the greasy luster on the outer surface. The former explanation is more plausible because often materials that are not lustrous will be recovered from the same location as lustrous materials. If soil conditions were responsible for the luster, then all specimens should be affected.

In examining a representative sample of flaking debris or artifacts that are suspected of being altered, an investigator should find a number of specimens which exhibit a relict dull area surrounded by extreme vitreousness. This situation suggests that the dull area has not

been flaked subsequent to heating whereas the vitreous area has been (see Figure 19). Soil conditions would not produce this type of differential preservation.

Color change which occurs at a lower temperature than the significant change resulting in a greater ease in flaking, may be considered a reliable indication if accompanied by vitreousness.

One other suggestion is appropriate. If local outcrop materials differ in texture from site specimens, it is a simple task to heat the local materials, fracture them, and determine if the resulting surface resembles site materials. This is how Mr. Crabtree first suspected the thermal method had been employed (see Literature Review).

Figure 19.--Specimens from the University of Florida Collections Showing Dull Areas Not Flaked Subsequent to Thermal Alteration Surrounded by Extreme Vitreousness in Areas that Have Been Flaked after Alteration.

SUMMARY AND INTERPRETATION

Despite derogatory comments by experimenters who have "proved" that subjecting flint to fire has only destructive effects (e.g., Pond 1930; Ellis 1940), enough firsthand accounts exist (e.g., Schumacher 1877; Powers 1877; Man 1883) to warrant investigation of the technique.

Extensive experiments conducted throughout a two-year period, in which flint materials were exposed to heat under numerous diverse conditions, have demonstrated that the alteration of siliceous rocks, when critical temperatures are reached slowly and maintained for a sustained period, probably conferred an advantage to prehistoric man in manufacturing his chipped stone implements.

The oft-quoted method of dripping cold water on hot rocks has been largely responsible for discrediting fire as a contributing factor in flintworking technology. Attempts by this investigator to "chip" with cold water produced no flakes at all and resulted in a crazing of the rock thus confirming the findings of Pond (1930) and Ellis (1940). However, if one returns to the reports of Schumacher (1877), Powers (1877), and Man (1883), they report removal of rocks from a hot fire after which they are shaped into implements. The only objection to these accounts is their brevity. A more detailed description or closer observation

by those authors may have revealed that the stones were not subjected to an open fire, that they may have been buried, or that they may have been allowed to remain in the heating environment for an extended period of time. My investigations have shown that:

1. Materials removed from a hot oven did not fracture when exposed immediately to a cool environment if the temperature necessary to effect an alteration in the rock had been reached slowly and maintained for a long enough period for the process to be completed.

2. Under point tensile load, a greater reduction in strength occurs when the rock materials are removed immediately from a hot oven to a cool environment. Limited tests gave results as follows: materials heated to 350°C but allowed to cool in the oven had a 40% loss in strength over the unheated control. A sample from the same nodule which had been heated to 350°C and exposed immediately to a cool environment had a 63% loss in strength--an additional reduction of 23%. It is possible that for certain extremely inelastic rock types exposure to these increased stresses was desirable to make chipping easier.

The accounts of the above-mentioned reporters are probably accurate as far as they go. Their descriptions, perhaps, have been misinterpreted more because they are incomplete rather than incorrect.

Another objection which might be raised with regard to primitive man's heating of rocks would be that since he had no thermometer, he had no way of knowing how high the

temperature was. This would be almost as ridiculous as saying that he had no conception of time because he hadn't invented the watch. Besides, these investigations demonstrated that, at least for Florida cherts, temperatures between 350°C and 600°C (a range of approximately 500°F) would effect a desirable alteration in the material. That is, as long as the elevation of temperature was gradual, there was no increased ease of chipping at 600°C over that of 350°C. While there is no apparent increase in flaking ease, however, quite often attempts to chip flint materials that had been heated to 500°C-600°C or had been heated to 350°C-400°C and removed immediately from the hot oven, resulted in a lateral snap due to end shock. Thus, there is additional strength loss with increased temperatures or increased stress. This fact was supported by point tensile strength tests. Below 300°C a satisfactory change did not occur in Florida cherts. Materials were not tested above 600°C (approximately 1100°F) because the aborigines probably could not reach or sustain such high temperatures.

It remains to determine if the three major problems to be investigated in this study have been successfully solved.

1. A desirable change does occur when Florida cherts are thermally altered resulting in a stone that is easier to flake than its unheated counterpart. No structural change occurs in that the size, shape, and orientation of the individual microcrystals remain the same, but through the removal of interstitial water, the microcrystals are fitted

closer together when certain materials other than SiO_2 serve as fluxes. When the flaw is introduced which is preliminary to and necessary for fracture to occur, the heated rock responds more like glass than a rock aggregate. In other words, crystal boundaries are no longer interfering with the removal of flakes. These statements have been substantiated throughout this dissertation by rock mechanics tests, scanning electron microscope illustrations, analyses demonstrating a reduction in surface area of heated materials, as well as intuitive observations and experiments.

2. Prehistoric peoples were aware of the advantages conferred by thermally altering their lithic materials because the chipping debris recovered from archaeological sites has been intentionally flaked following subjection to heat. This is apparent because the flakes have bulbs of percussion which demonstrate that there has been a point of impact. Bulbs of percussion are not exhibited if rocks explode when too rapid heating or cooling occurs. Instead, there are "potlid" fractures or conchoidal fractures which do not show a point of impact. The flaked surface of the altered specimens are extremely vitreous, varying appreciably from outcrop samples of the same materials. Very often also, due to the presence of minute amounts of iron, there is a color change from grey-beige-brown to pink-red. While color change occurs at a lower temperature than the significant change resulting in easier flaking and will not occur at all unless iron is present, it may be considered a reliable factor if used in conjunction with vitreousness.

A review of available literature has uncovered numerous accounts of the use of fire as an aid in the chipping process--at least enough to warrant the conclusion that primitive peoples knew that fire was capable of producing a desirable change in flint materials.

3. The problem of recommending an easy test to determine if materials from archaeological sites have been intentionally altered has not been very rewarding. Test after test has not suggested any really reliable criteria. From an examination of a representative sample of flaking debris, however, an investigator should find a number of specimens exhibiting a relict dull area surrounded by areas of extreme vitreousness. This situation strongly suggests that the dull area has not been flaked subsequent to heating whereas the vitreous areas have been.

The evidence presented in this report leads to the conclusion that the manufacture of chipped stone implements is easier to execute if lithic materials are first cautiously subjected to critical temperatures (350°C-400°C for Florida cherts) for sustained periods (12-24 hours but varying with sample size). After nearly two million years of experimentation with fire and stone, it should come as no surprise to find that primitive peoples were well aware of the advantages of thermal alteration.

Artifacts have always been the archaeologist's best friend. A recognition of textural changes occurring when siliceous materials are thermally altered provides knowledge relating to lithic technology and primitive man's behavioral patterns, thus establishing a new dimension to archaeological interpretation.

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BIOGRAPHICAL SKETCH

Barbara Ann Purdy was born August 23, 1927 at San Diego, California. In June, 1945, she was graduated from Herbert Hoover High School. In June, 1948, she received the degree of Bachelor of Arts with a major in Zoology from San Diego State College. In 1949, she was employed at Scripps Institution of Oceanography at La Jolla, California in the Department of Marine Microbiology. From 1949 to 1953, she was employed in various departments on the campus of the University of California at Davis. From 1960 to 1965 and intermittently to 1967, she typed theses and served as Thesis Editor at Washington State University, Pullman, Washington. In the fall of 1964, she enrolled in the Department of Anthropology at Washington State University. From 1965 to 1967, she was a Research Assistant and received the Master of Arts degree in 1967. In 1967, she enrolled in the Department of Anthropology at the University of Florida in order to pursue work leading to the degree of Doctor of Philosophy. In July, 1969, she attended a four-week flint-working session at Shoshone Falls, Idaho sponsored by Idaho State University with funds granted by the National Science Foundation. She was a teaching assistant from 1968 to 1970 in the Department of Anthropology and taught American Institutions in the University College at the University

of Florida during the fall quarter, 1970 and the winter quarter, 1971.

Barbara Ann Purdy is married to Laurence Henry Purdy and is the mother of four children. She is a member of the American Anthropological Association and the Society for American Archaeology.

I certify that I have read this study and that in my opinion it conforms to acceptable standards of scholarly presentation and is fully adequate, in scope and quality, as a dissertation for the degree of Doctor of Philosophy.

Charles H. Fairbanks

Charles H. Fairbanks, Chairman
Professor of Anthropology

I certify that I have read this study and that in my opinion it conforms to acceptable standards of scholarly presentation and is fully adequate, in scope and quality, as a dissertation for the degree of Doctor of Philosophy.

Wm. R. Bullard jr.

William R. Bullard, Jr.
Associate Curator, Florida State Museum

I certify that I have read this study and that in my opinion it conforms to acceptable standards of scholarly presentation and is fully adequate, in scope and quality, as a dissertation for the degree of Doctor of Philosophy.

Theron A. Nuñez Jr.

Theron A. Nuñez, Jr.
Associate Professor of Anthropology

I certify that I have read this study and that in my opinion it conforms to acceptable standards of scholarly presentation and is fully adequate, in scope and quality, as a dissertation for the degree of Doctor of Philosophy.

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This dissertation was submitted to the Dean of the College of Arts and Sciences and to the Graduate Council, and was accepted as a partial fulfillment of the requirements for the degree of Doctor of Philosophy.

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